HIGHWAY RESEARCH RECORD

Number 412 | Nuclear Applications, Pipe Corrosion, and Aggregate and Glass Bead Testing

7 reports

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Number	Nuclear Applications,
412	Pipe Corrosion, and Aggregate
	and Glass Bead Testing

7 reports
prepared for the
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Although these reports are not submitted for approval to the Academy membership or to the Council of the Academy, each report is reviewed and processed according to procedures established and monitored by the Academy's Report Review Committee.

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FOREWORD

This RECORD contains 4 papers on nuclear applications to highways, 1 paper on corrosion of metal pipe, 1 paper on aggregates, and 1 paper on glass beads in paint.

Hughes describes laboratory and field evaluation of a nuclear asphalt-content gauge that showed a laboratory precision of 0.06 percent asphalt.

An instrument for measuring concentrations of hydrocarbon, CO, and NO_x pollutants in automobile exhausts is the subject of Goodman's paper.

The paper by Duffey et al. deals with the analysis of concrete using a 100-μg source of californium-252. The mixes contained 3 different coarse aggregates: quartzite gravel, limestone, and diabase. This neutron-capture gamma ray technique has possible applications in nondestructive determination of concrete components and in concrete quality control.

Lepper et al. report on their use of neutron radiation to measure the water content of fresh concrete.

A Kansas study of the service life and corrosion of nearly 2,000 corrugated metal pipes, some as old as 43 years, is reviewed in the paper by Worley and Crumpton.

Work done in the Federal Republic of Germany by Kohler and Nagel compares the Los Angeles, Deval, German impact, British impact, and modified Marshall impact methods of testing small-sized coarse aggregates. A relative reproducibility of as much as 20 to 30 percent was found in comparisons of similar tests performed in different laboratories.

Crumpton and McCaskill discuss the manner in which various items of laboratory equipment were used in studying the performance of glass beads in paint.

This assortment of reports will be of interest primarily to materials engineers and materials researchers.

EVALUATION OF A NUCLEAR ASPHALT-CONTENT GAUGE

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The Troxler asphalt-content gauge, model 2226, was evaluated in the laboratory and taken into the field where its results were compared to conventional reflux values. The precision as evaluated in the laboratory was found to be excellent, equivalent to 0.06 percent asphalt with a 45-sec count. The gauge has to be recalibrated for different aggregates and, as a practical matter, should be recalibrated for different mix types. Sample preparation is important, and the samples should be as uniform as possible. The accuracy appears to be as good as that of the reflux extractor.

●THERE have been several reports (1, 2, 3) on the use of nuclear gauges to measure the asphalt content of bituminous mixes. The earliest report (1) was in 1956 and was on research in which experimental equipment was used. That report concluded that, although the theoretical principles involved had been experimentally validated, the variability of the results and the cost of the equipment precluded the use of the apparatus at that time. More recent reports have dealt with the use of commercially available nuclear moisture gauges to measure asphalt content; that is possible because the measurements of both moisture and asphalt content are based on the detection of thermalized neutrons. However, because equipment adaptation is necessary and the variability of the test results is rather large, this equipment has not been widely accepted for measuring asphalt content.

More recently gauges designed expressly for the purpose of measuring asphalt content have become commercially available (4, 5). In 1969 the author (6) reported on the use of such a gauge. It was reported that the precision of the gauge, about 0.20 percent asphalt, was fairly good but that a relatively long counting time, 18 min, was required. Also the accuracy left something to be desired; the correlation between count rate and asphalt content had a standard error of 0.30 percent asphalt. It was suggested that the gauge could be redesigned to improve its accuracy, and this conclusion was generally substantiated by a study done at the University of Southwest Louisiana (7). The manufacturer, Troxler Electronic Laboratories, agreed with this conclusion and made a second-generation gauge available in November 1969.

This report is essentially concerned with an evaluation of the redesigned Troxler gauge, model 2226, shown in Figure 1. The operation of the gauge is similar to that of the original model in that the sample pan is filled and inserted into a drawer for testing. However, it is different in 2 important respects. One is that it operates on the basis of direct transmission rather than backscatter. This means that the sample is placed between the source (300 mCi Am Be) and the He_3 detector tubes, which are much more efficient than the BF_3 tubes previously used. This feature minimizes the influence of the location of the asphalt, a serious drawback in the original gauge. The other difference is the inclusion of a self-standardizing operation (Fig. 2) that converts the count obtained from the scaler into a count ratio. This feature simplifies the gauge operation tremendously by eliminating the requirement for a separate standard count.

PURPOSE AND SCOPE

The purpose of this evaluation was to determine the precision of the Troxler asphalt-content gauge and to evaluate its accuracy under several variables. A laboratory eval-

uation of the gauge constituted the initial phase of the project. The precision was determined by performing several repeat tests on the same sample, and the accuracy was evaluated by analyzing the effects of several variables. More specifically, in the latter instance, it was necessary to determine for which variables the gauge must be recalibrated. The variables investigated were aggregate type and gradation, asphalt content and asphalt penetration, and producer. On the basis of the author's previous study it was anticipated that different aggregates would produce different count rates, and this expected result was checked by using 4 aggregates: granite, limestone, greenstone, and gravel.

Although the previous study had indicated no effect from gradation, it was thought that gradation should be included as a variable. The gradations studied are given in Table 1. These variables were included in an experimental design to cover an asphalt-content range of 0 to 7 percent as given in Table 2 (all asphalt contents were calculated on a percentage by weight basis); Table 2 also gives the producer and penetration grade. All mixes were of sufficient weight to allow 2 samples of 6,700 grams each to be tested. The laboratory phase of the study was followed by a field-testing program.

NUCLEAR TESTS

The precision of the gauge was established by performing 30 three-minute (1-position) repeat tests on the same sample. On a 6 percent fine limestone mix the standard deviation was 130 counts (equivalent asphalt content = 0.06 percent), which provided a variation coefficient of 0.27 percent. These results are shown in Figure 3, which also shows that count rate is independent of temperature from the normal mixing temperature of 280 F to 140 F. Based on these data and a 45-sec (0.25 test position) count rate, it was determined that for a 45-sec count the precision, or ability of the gauge to repeat a measurement, would have a 95 percent confidence limit of 0.12 percent asphalt.

The accuracy of the gauge under the previously mentioned variables was evaluated by employing 2 statistical techniques. More specifically, these techniques were used to determine which variables would likely require the establishment of separate calibration curves. The first technique employed was a regression analysis of mixes 1 through 48 (Table 2). An indication of the influence of both asphalt penetration and producers was gained by performing an analysis of variance. These analyses, as well as one between design and extracted asphalt content and one dealing with field calibration and testing, are discussed in the following sections.

Sample Preparation

Sufficient material was used in all of the mixes to allow tests on 2 pans for each mix so that an indication of "between-pan" variation could be obtained. The first tests were performed on a fine-gradation mix, and the difference between the 2 test pans was much greater than had been found for the "within-pan," or precision, data that had been obtained. This led to an investigation of the sample-preparation techniques, which revealed much greater reproducibility when a mechanical sample splitter was used than when the sample was split by hand. This difference is given in Table 3. The average values for the difference between pans was about 0.22 percent for hand and 0.04 percent for mechanical splitting. This difference pointed out the necessity for preparing samples with a sample splitter. It also emphasized that differences in asphalt content can be caused by a relatively small amount of segregation, even in a fine-graded mix. Thus, as has been recognized in the past, sample preparation is quite important; because it does cause single test values to vary widely, sample averages should be used as extensively as practicable.

Aggregate Effect

The influence of aggregate type and gradation was determined by performing linear regression analyses on each aggregate for each gradation. All gradations were then pooled, and a regression analysis was made for each aggregate. As mentioned earlier, the initial study had substantiated clearly the theoretical principle that separate cali-

Figure 1. Gauge with sample drawer open.

Figure 2. Gauge operation.

Table 1. Mix gradations.

Item	Coarse (percent passing)	Medium (percent passing)	Fine (percent passing)
Sieve size			
1½ in.	100		
¾ in.	75	100	
½ in.			100
⅜ in.		70	90
No. 4	40	50	60
No. 8	30	35	45
No. 30			
No. 50		10	15
No. 100		6	10
No. 200	3		6
Asphalt content	0 to 5	0 to 6	0 to 7
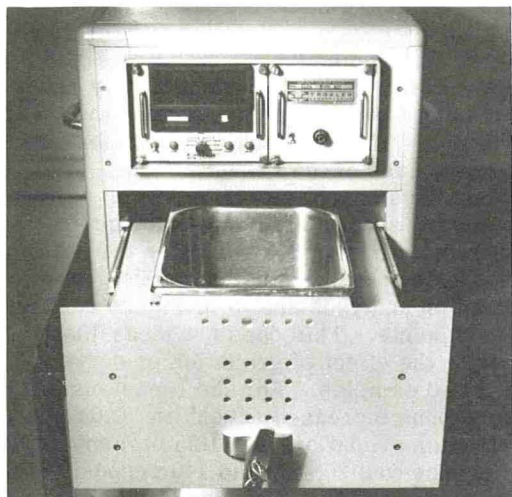
Table 2. Mixes tested.

Mix[a]	Aggregate	Gradation	Asphalt Content (percent)	Asphalt[b]
1 to 4	Granite	Coarse	0, 3, 4, 5	1 (85 to 100)
5 to 8	Granite	Medium	0, 4, 5, 6	1 (85 to 100)
9 to 12	Granite	Fine	0, 5, 6, 7	1 (85 to 100)
13 to 16	Gravel	Coarse	0, 3, 4, 5	1 (85 to 100)
17 to 20	Gravel	Medium	0, 4, 5, 6	1 (85 to 100)
21 to 24	Gravel	Fine	0, 5, 6, 7	1 (85 to 100)
25 to 28	Limestone	Coarse	0, 3, 4, 5	1 (85 to 100)
29 to 32	Limestone	Medium	0, 4, 5, 6	1 (85 to 100)
33 to 36	Limestone	Fine	0, 5, 6, 7	1 (85 to 100)
37 to 40	Greenstone	Coarse	0, 3, 4, 5	1 (85 to 100)
41 to 44	Greenstone	Medium	0, 4, 5, 6	1 (85 to 100)
45 to 48	Greenstone	Fine	0, 5, 6, 7	1 (85 to 100)
49	Gravel	Medium	5	1 (85 to 100)
50	Gravel	Medium	5	2 (85 to 100)
51	Gravel	Medium	5	1 (60 to 70)

[a]Each mix was split and duplicate samples run for each mix.
[b]1 = Esso; and 2 = Chevron.

Table 3. Effect of type of splitting.

Splitting Method	Pan 1	Pan 2	Difference Count Rate	Difference Asphalt Content (percent)
Hand	9,658	9,574	84	0.17
Hand	12,840	12,974	134	0.27
Mechanical	9,543	9,531	12	0.02
Mechanical	12,191	12,214	23	0.05

Table 4. Effect of gradation.

Aggregate	Gradation	Slope	Intercept	Standard Error	Correlation Coefficient
Granite	Coarse	1,868	38,544	0.09	0.9994
	Medium	1,896	38,744	0.18	0.9985
	Fine	1,883	38,907	0.13	0.9994
	Pooled	1,902	38,662	0.14	0.9985
Limestone	Coarse	2,026	38,502	0.19	0.9973
	Medium	2,177	37,822	0.20	0.9981
	Fine	2,209	38,213	0.15	0.9992
	Pooled	2,173	38,090	0.20	0.9972
Greenstone	Coarse	1,953	43,366	0.18	0.9976
	Medium	1,873	43,850	0.07	0.9997
	Fine	1,921	44,075	0.17	0.9990
	Pooled	1,940	43,665	0.19	0.9974
Gravel	Coarse	1,885	38,964	0.25	0.9955
	Medium	2,091	38,919	0.39	0.9926
	Fine	1,971	39,797	0.22	0.9984
	Pooled	2,038	39,040	0.33	0.9918

Figure 3. Precision results and lack of temperature influence on count rate.
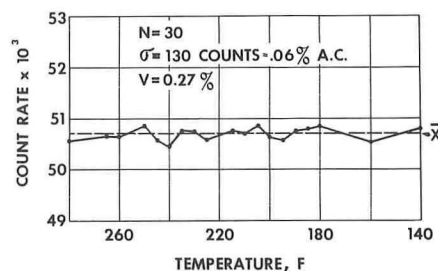
bration curves would be needed for each aggregate. Table 4 gives the slopes and intercepts from the linear regression analyses as well as the standard error and correlation coefficient for each gradation and for each aggregate. Of primary importance initially are the very high correlation coefficients obtained under all gradations and aggregates; all are more than 0.990, which indicates that count rate is definitely related to asphalt content. Also of importance is the standard error, which indicates the level of accuracy that can be expected from the prediction. The standard error values are generally 0.20 percent asphalt or less, except in the case of the gravel mixes.

Data given in Table 4 also show that gradations have essentially no effect on calibration or, more precisely, on count rate. The standard errors and correlation coefficients for the individual gradations are not sufficiently improved over those for the pooled values to warrant the use of the former. This same conclusion can be drawn from the graphical representations of the regression analyses shown in Figure 4. There is no discernible difference between the gradation points. This does not mean that there is no gradation effect. As stated previously, the effect of gradation or segregation was apparent from tests on supposedly identical samples. For the limestone gradation, for example, the average difference between pans increased sevenfold: 0.04 percent in the fine gradation, 0.10 percent in the medium gradation, and 0.28 percent in the coarse gradation. The average count-rate difference was 20, 40, and 140 respectively. Naturally, this phenomenon is not unique with nuclear testing. However, the ability to retest the same sample and the speed of testing with the nuclear method make the differences much more apparent.

The results from pooled aggregate analyses (Table 5) show clearly by the variation in intercepts that separate calibration curves are necessary for each aggregate. Although it does not appear that a change in gradation requires a change in calibration, as a practical matter a change in gradation is normally accompanied by a change in aggregate type. This means that as a practical matter a change in gradation should necessitate at least a recheck on the calibration. This subject will be discussed in more detail later.

Because the slopes between aggregates are reasonably close, it was thought it would be possible to use only a 0 percent, or dry aggregate, point and the average slope of 2,013 counts versus percentage of asphalt content to establish a reasonably accurate calibration curve. Predictions based on this method did not compare well with the actual asphalt contents, and this method was dropped from further consideration.

Asphalt Effect

Whether asphalt producer or asphalt penetration affected the count rate was determined by testing 3 mixes (49, 50, and 51). The mixes had a single gradation, aggregate type, and asphalt content and varied only in penetration or producer. An analysis of variance indicated that statistically there was a significant difference between asphalts, and in this case the difference appeared to be due to penetrations. The average count rate and the equivalent asphalt contents measured are as follows:

Mix	Count Rate	Asphalt Content (percent)
49	49,268	5.00
50	49,280	5.01
51	49,024	4.88

Although there may have been some statistically significant difference attributable to penetration, it appeared to be reasonably small and can be accommodated, it is believed, in the field calibration.

CONVENTIONAL TESTS

A basis was established for comparing nuclear asphalt-content values and conventional reflux values by extracting 36 of the first 48 mixes (12 were dry or 0 percent

asphalt mixes) by the reflux method and correlating the values with the design asphalt content. Figure 5 shows the regression line and the pertinent statistical information. As expected, the correlation coefficient was high (0.994) and the slope was almost unity (1.02). But the average extracted asphalt content was 0.10 percent lower than the design average, which indicated a bias in the method. That bias in extracted asphalt content was not unexpected because it is quite often found that the amount extracted is not so high as that put into the mix. The bias should be considered whenever it is desired to correlate nuclear values and extracted values because the asphalt content cannot be controlled as well in the plant as in a laboratory.

The standard error of the conventional values versus design values was 0.13 percent as compared to generally less than 0.20 percent for the nuclear correlations given in Table 4. Because of the great speed advantage in the nuclear method, the slight loss in accuracy should be more than compensated for by making more tests.

FIELD CALIBRATION

The primary criteria for establishing a field calibration procedure were that it must be technically sound and be practical. For the first criterion, the following guidelines were established:

1. Each aggregate must be calibrated separately;
2. At least 2 points are necessary to establish a calibration curve;
3. Each mix type must be checked for calibration; and
4. Asphalt type should be checked periodically for calibration.

Guidelines for the second criterion were as follows:

1. The procedure must be one that plant personnel can master; and
2. The calibration procedure must not be lengthy or be required too often.

With these guidelines it was decided to calibrate as soon as possible after starting up a plant. Because moisture variations affect the count rate of the gauge, the ideal sampling point appeared to be the hot bin, where the moisture content should be reasonably stable. This was also advantageous because the aggregate, after it was blended in the proper proportions, could be tested dry to establish the 0 percent point on the calibration curve and then, because it was still hot, could be mixed with an asphalt sample from the storage tanks to produce a second point on the calibration curve near the optimum asphalt content for that mix. With this procedure, about 1 hour is required for establishing a calibration curve; afterward, testing can commence. The main parameter established by this process is the slope of the curve because the intercept will change from time to time depending on the moisture in the aggregate. Therefore, the 0 percent point only should be checked at least once a day and more often if variable moisture conditions exist in the aggregate stockpiles.

INITIAL FIELD TESTING

The procedure just given was used to check 5 plants during the fall of 1970. Table 6 gives the results of the nuclear tests using both 1 and 0.25 position counts and of the corrected reflux tests. The correction values are the differences between the design asphalt content used in making up the calibration sample and the amount extracted by the reflux test. This procedure is in agreement with results of the correlation in the preceding section on conventional tests. The comparison between nuclear and corrected reflux averages is very good; the average difference for all results is only 0.13 percent asphalt for the 1 position and 0.09 percent asphalt for the 0.25 position.

EXTENDED FIELD TESTING

Throughout the 1971 construction season, the asphalt-content gauge was used at an asphalt plant supplying base, intermediate, and surface mixes for an Interstate project. A technician from the Virginia Department of Highways operated the gauge in conjunction with the conventional reflux method used for acceptance purposes. Whenever a conven-

Figure 4. Regression analysis for aggregates.

Figure 5. Regression analysis of asphalt.
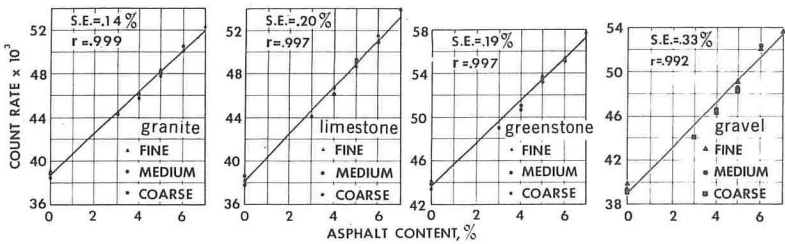
Table 5. Effect of aggregate.

Aggregate	Slope	Intercept	Stand. Error
Granite	1,902	38,662	0.14
Limestone	2,173	38,090	0.20
Greenstone	1,940	43,665	0.19
Gravel	2,038	39,040	0.33
Average	2,013		

Table 6. Results of initial field testing.

Plant	Cali-bra-tion	Mix	Num-ber of Tests	Nuclear 1 Position Avg.	Nuclear 1 Position Standard Deviation	0.25 Position Avg.	0.25 Position Standard Deviation	Corrected Reflux (percent) Avg.	Corrected Reflux (percent) Standard Deviation	Corrected Asphalt Content (percent)
1	1	Base	10	4.82	0.48	4.87	0.48	4.85	0.40	0.50
1	2	Base	8	5.31	0.71	5.32	0.64	5.19	0.56	0.84
2	1	Surface	10	5.70	0.22	5.69	0.17	5.74	0.14	0.28
2	2	Surface	10	5.86	0.37	5.78	0.28	5.78	0.14	0.16
3	1	Interme-diate	10	4.60	0.20	4.57	0.14	4.57	0.12	0.19
3	2	Interme-diate	6	4.29	0.15	4.21	0.23	4.43	0.15	0.05
3	3	Surface	6	4.53	0.11	—	—	4.74	0.15	0.17
4	1	Surface	10	5.51	0.44	5.61	0.47	5.72	0.37	0.11
5	1	Base	8	3.96	0.31	—	—	4.16	0.21	0.22
5	2	Interme-diate	7	5.12	0.49	—	—	4.96	0.24	0.14

Table 7. Results of extended field testing.

Mix	Nuclear Difference From Job Mix	Nuclear Standard Deviation	Nuclear Correction	Nuclear Number of Samples	Corrected Reflux Difference From Job Mix	Corrected Reflux Standard Deviation	Corrected Reflux Correction	Corrected Reflux Number of Samples
Base								
Before	0.5	0.4	—	61	0.0	0.3	0.2	37
After	-0.1	0.2	—	67	-0.1	0.2	—	67
Intermediate	0.0	0.4	—	64	0.0	0.4	—	64
Surface	0.2	0.2	—	25	0.2	0.2	—	25

Table 8. Results of hot-bin and washed-sample analyses.

Mix	Sieve (percent passing) 1 in.	3/4 in.	3/8 in.	No. 4	No. 8	No. 30	No. 50	No. 200	Number of Samples
Base									
Hot bin	100.0	75.7	—	41.8	26.0	—	—	3.4	19
Reflux	100.0	75.1	—	44.0	28.8	—	—	5.3	20
Intermediate									
Hot bin	100.0	84.3	76.2	47.2	28.3	—	6.3	3.8	24
Reflux	100.0	84.6	76.4	46.1	29.1	—	8.0	5.0	26
Surface									
Hot bin	—	—	95.5	61.1	38.7	16.2	9.8	2.9	6
Reflux	—	—	95.1	60.7	41.2	20.9	13.4	4.2	6

tional sample was tested, a comparison nuclear sample was tested. However, because of the speed of nuclear tests, many more of those tests were made. The comparative tests provided an opportunity to evaluate the gauge under everyday plant conditions and by a technician not familiar with it.

From the data obtained under those conditions, it was found that testing the aggregate dry to establish the 0 percent calibration point daily was probably not necessary. The standard deviation determined from samples taken daily during a period of several months was only 190 counts, or approximately equivalent to 0.12 percent asphalt. On the basis of these results, it is anticipated that two 0 percent calibration points per week will suffice.

A sample preparation effect related to aggregate gradation, heretofore not evident, appeared when the base mix was tested; because it was reasonably coarse-graded, it often had a coarse surface texture. Table 7 gives the results for the comparative tests obtained from this project. The "before" data for the base mix are the results before any consideration was given to the surface texture of the sample. The nuclear asphalt contents averaged 0.5 percent more than the job-mix value, and the corrected-reflux results averaged the same as the job-mix value. The standard deviations for the 2 types of tests were comparable, which indicated that the nuclear values were as consistent as those of the reflux. Therefore, the nuclear readings were higher than should be expected.

After it was ascertained that the calibration was accurate and not responsible for the high values, it was noticed that the finished texture in the pan was always coarse when a high value was obtained. Particular attention was then paid to placing most of the coarse aggregate in the sample pan first, thus providing a relatively smooth-finished texture in the pan. The results became lower and consistent with the corrected-reflux values as shown by the "after" data for the base mix.

For the other mix types, it is obvious from the data given in Table 7 that the nuclear results can estimate the job-mix asphalt content as closely as, if not closer than, the reflux method. On this project, there appeared to be somewhat more variability in the nuclear method than was found from previous testing. However, because the method is appreciably faster than the conventional one, many more tests can be run and the testing variability effectively reduced.

QUICK-GRADATION ANALYSIS

The adoption of a rapid-test method for the acceptance of asphalt content requires a reevaluation of the method of acceptance for the gradation of asphalt-concrete mixes. Having to wait for a gradation obtained from a washing process negates some of the advantage of the speed of the nuclear asphalt-content test. It appears that the best way of speeding up gradation tests would be through the use of hot-bin samples. This procedure was used on the field project as a comparison with the gradation values from the reflux tests. The results are given in Table 8.

There appears to be very good agreement between the 2 methods, except in the fine-sieve sizes. Because the preparation of hot-bin samples requires splitting the sample and combining it proportionately, some fines are lost. However, this method still appears to offer some hope for speeding up the gradation analysis.

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MEASUREMENT OF AUTOMOBILE EXHAUST POLLUTANT CONCENTRATIONS BY USE OF SOLID KRYPTONATES

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ABRIDGMENT

●IT is hardly necessary to point out, in the current climate, the importance of automobile exhausts as contributors to air pollution. Inspection of both new and used cars will be necessary to ensure that, at least at periodic intervals, the emissions from a particular automobile do not exceed prescribed levels. Neither plans nor mechanisms for implementation of such an inspection program are yet established in their entirety, but some features are nevertheless clear. Suitable instrumentation for the measurement of automotive pollutant emission levels must be available and be operable by nontechnical personnel, particularly for used-car inspections. The instrumentation may be installed in fixed locations, such as inspection lanes, or may be operated for on-the-spot inspections by highway patrol cars and the like. The instrumentation should, therefore, be portable. Finally, automotive repair personnel must have instrumentation available at reasonably low cost to inform them that their repairs and adjustments have been effective.

Reasonably sophisticated instrumentation exists for the measurement of CO and hydrocarbon pollutant concentrations. Generally, these are single-channel instruments. An instrument suitable for the measurement of NO_x is lacking. An instrument having the characteristics of simplicity, multichannel capability, portability, and low cost does not exist.

This paper describes the development, still in progress, of an instrument suitable for use in an inspection program whose implementation is projected for 1975.

GENERAL DESCRIPTION

The instrument under development uses not only solid-state components but also solid-sensing materials. These sensing materials, termed Kryptonates, are solid substrates into the surface of which the radioisotope Kr^{85} has been stably incorporated. Their preparation, properties, and numerous application areas have been described (1, 2, 3). All substrates used for this instrument have been prepared from small particle-sized powders, which were placed in a pressure vessel wherein Kr^{85} was diffused into the surface layers at high temperatures and pressures.

Kryptonates, after preparation, can be stabilized for use at all temperatures below their stabilization temperature (1). The retained Kr^{85} is released from the solid by reaction with the pollutant gas to be detected. The release rate of Kr^{85} is dependent on the surface reaction rate and, hence, on the pollutant concentration. The release rate of Kr^{85} resulting from reaction with the solid Kryptonate can be measured by use of a counting chamber observed by a Geiger-Müller (GM) tube.

It is possible to prepare Kryptonates from almost any solid. Judicious selection of the host solid for the Kryptonate can be made so that reaction takes place only with the pollutant to be detected. Specificity can thus be attained and a multichannel instrument constructed by use of appropriately selected sensing substrates. High sensitivity is achieved by virtue of 2 other properties of Kryptonates. Kr^{85} trapped by the solid is located only in the surface layers of the solid. Only that reaction taking place at the

surface of the solid plays a part in the gaseous detection. Further, the high sensitivity for detection of radioisotopes that is provided by conventional techniques is utilized.

Kr^{85} is one of the safest and most convenient radioisotopes known. It emits, almost exclusively, a beta particle that is easily shielded. Its half-life is more than 10 years, making corrections for activity decay negligible. Because it is a rare gas, it does not enter into human metabolic processes, and significant accumulation in body tissues should not occur.

SENSING SUBSTRATES

Suitable sensing substrates were selected (4) for the detection of automobile exhaust pollutants, specifically hydrocrabons, carbon monoxide, and NO_x. The substrates were responsive to the individual pollutants and nonresponsive to other pollutants present in the exhaust gases. A breadboard instrument was constructed, and the characteristics of these sensing materials were evaluated.

The breadboard instrument currently utilizes an I_2O_5 Kryptonate sensor operated at 350 F. A maximum sensitivity of about 80 ppm CO has been achieved in the breadboard instrument. The Kr^{85} release rate is linear with CO concentration as shown in Figure 1.

For the detection of hydrocarbons, a PtO_2 Kryptonate operating at 500 F is used. The Kr^{85} release rate is a linear function of concentration of a particular hydrocarbon in the gas stream but is a function of the hydrocarbon molecular weight as shown in Figure 2. The response is dependent on the number of $-CH_2-$ groups present in the linear hydrocarbon. For the breadboard instrument, the maximum sensitivity was about 0.5 ppm of hexane.

The sensor utilized for NO_x is a hydroquinone clathrate. At room temperature, the clathrate responds to NO_2 but not to NO so that it is necessary to convert the NO pollutant to NO_2 in order to detect it. The breadboard instrument currently uses a room-temperature catalyst for the NO to NO_2 conversion. The maximum sensitivity for the breadboard instrument is 1 ppm.

It should be emphasized that the maximum sensitivities listed for all sensors are not the maximum achievable sensitivities. For instance, the clathrate sensing material used to attain 1-ppm sensitivity had been diluted with non-Kr^{85} containing hydroquinone. These sensitivities are listed to demonstrate that much more sensitivity exists than is required if this instrument is used to monitor exhaust gases directly.

All sensing materials listed have been tested for cross interferences, and none has been detected except that the clathrate sensor responds to water vapor if the RH of the gas stream is more than approximately 90 percent. This interference is eliminated by dilution with a desiccated gas stream.

BREADBOARD INSTRUMENT

The breadboard instrument that has been constructed is shown in Figure 3, and the flow system contained therein is schematically shown in Figure 4. This breadboard instrument has a volume of 1.25 ft^3 and weighs 22 lb. Future versions are expected to be smaller and lighter.

This is a multichannel instrument designed to measure the concentration of each pollutant gas sequentially. The incoming automobile exhaust stream flows through a manifold. Solenoid valves direct gases through 1 of the 3 sensing chambers containing a Kryptonate sensor. The released Kr^{85} then passes through the counting chamber and is exhausted to the atmosphere through a pump. Flow rates through the instrument are approximately 1 liter/min. The Kr^{85} betas in the counting chamber generate pulses in the GM tube that are fed to a simple rate-meter circuit whose output is displayed on a meter. Two rate-meter time constants are available for selection: one short-time constant to permit more rapid approximate readings and one longer time constant to permit more accurate count rate readings. For the longer time constant, decay statistics yield an uncertainty of ±1.7 percent for a full-scale reading. The breadboard instrument has been designed to provide approximately half-scale deflection for pollutant concentrations corresponding to the projected 1975 standards that have been taken as 50, 5,000, and 250 ppm for hydrocarbons, CO, and NO_x respectively. The sensitivity can be easily reduced, however, by dilution with non-Kryptonate materials.

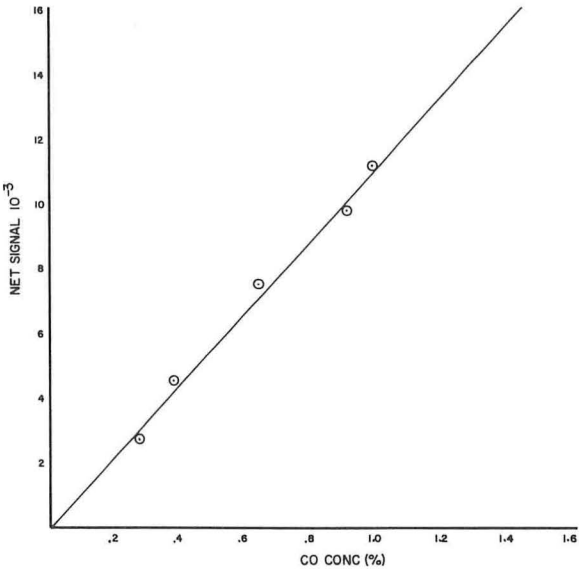Figure 1. Response of I_2O_5 Kryptonate sensor as a function of CO concentration.

Figure 2. Hydrocarbon species dependence of PtO_2 Kryptonate.
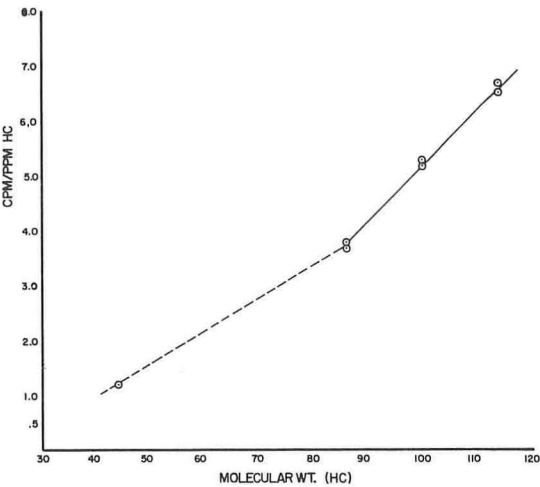

Figure 3. Breadboard instrument for monitoring automobile exhaust pollutants.
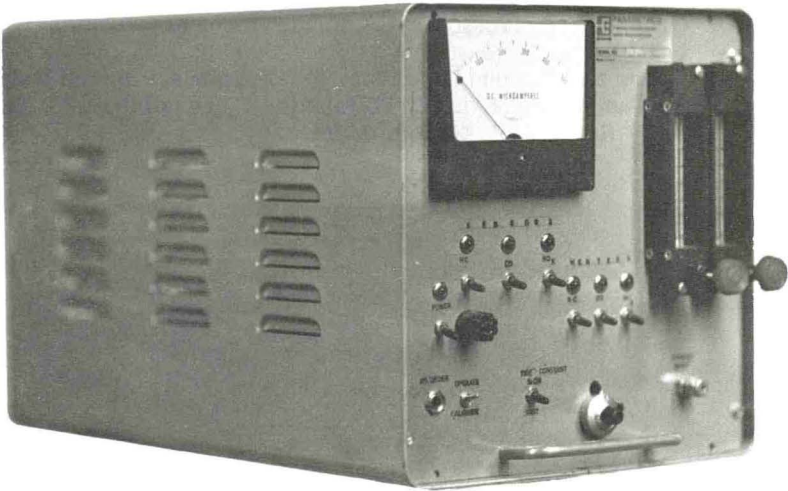
Figure 4. Flow diagram of breadboard instrument.
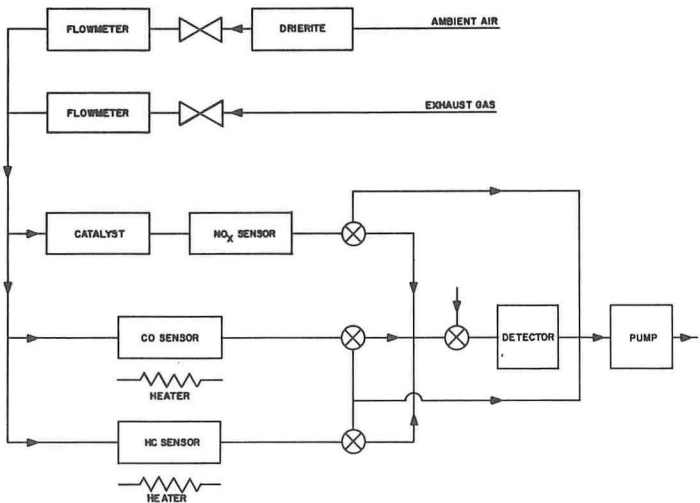
The breadboard was tested most thoroughly in our laboratory by the use of an exhaust stream from a 4-cycle lawn-mower engine. Indicated exhaust levels were compared with the measurements provided by an FID instrument for hydrocarbons and an NDIR instrument for CO. Satisfactory performance was obtained. Similar, but less extensive, testing with automobiles has been equally successful and promising. Poisoning of sensor response by continued exposure to exhaust gases was never observed, even with the use of leaded gasoline. Response times, measured from the time of application of a step change in pollutant concentration to the instrument inlet, are approximately 15 to 20 sec. It is expected that further design changes can reduce these response times.

Extensive testing has not, however, been performed as yet to determine the lifetime of individual sensor cells. It is calculated that each cell, containing 100 μCi of Kryptonate sensing material, could provide about 100 hours of monitoring time at an average concentration corresponding to a half-scale reading. During operation of the instrument, continuous exposure is not expected so that a 2- to 3-month operating life is projected. Such a cell would not require an individual license for the user.

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CONCRETE ANALYSIS BY NEUTRON-CAPTURE GAMMA RAYS USING CALIFORNIUM 252

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The feasibility of analyzing concrete and cement by a measurement of the neutron-capture or prompt gamma rays was investigated; a 100-μg californium-252 source was used to supply the neutrons. A lithium drifted germanium crystal detected the capture gamma rays emitted. The capture gamma rays from cement, sand, and 3 coarse aggregates—quartzite gravel, limestone, and diabase—were studied. Concrete blocks made from these materials were then tested. The capture gamma ray response of the calcium, silicon, and iron in the concrete blocks was in accord with the elements identified in the mix materials. The principal spectral lines used were the 6.42 MeV line of calcium, the 4.93 MeV line of silicon, and the doublet of iron at about 7.64 MeV. The aluminum line at 7.72 MeV was observed in some cases but at a lower intensity with the limited electronic equipment available. This nuclear spectroscopic technique offers a possible method of determining the components of sizable concrete samples in a nondestructive, in situ manner. In addition, the neutron-capture gamma ray technique might find application in control of the concrete and cement processes and furnish needed information on production operations and inventories.

●FROM THE point of view of the geochemical analyst, concrete may be considered as rock relocated and reformed at the convenience of the engineer. Because the amounts of concrete are large and the industrial value is high, monitoring the processes and the finished concrete is important. Nuclear techniques provide another avenue for determining the composition. Efforts to analyze concrete by measurements of the delayed gamma rays from the decay of neutron-activated products have been reported (1, 10). However, the gamma rays promptly emitted on neutron capture are also distinctive of an element but, because of detection difficulties, have been given less attention by analysts. Nevertheless, experiments to sense the gross constituents of rocks by capture gamma rays were reported some years ago (2).

More recently, the prompt gamma rays from the capture of neutrons from a nuclear reactor (3) and from californium-252 (4) have given information on the mineral values in geologic materials, e.g., gold, copper, nickel, titanium, and manganese. Consequently, this investigation on the capture gamma ray method has been extended to concrete made of typical aggregates. The results indicate that major constituents of formed concrete and its process components can be determined on sizable samples in a nondestructive manner with almost immediate results. The technique of using nuclear spectroscopy is described. The experiments performed suggest possible applications to the concrete industry.

PREVIOUS NUCLEAR STUDIES WITH CONCRETE

Density and moisture measurements by nuclear techniques have been investigated for concrete materials, such as aggregates (5, 6, 7, 8, 9). The density measurements have been made by transmission and backscattering, such as with the 0.66 MeV gamma rays from cesium-137. Moisture determinations have been made with neutron sources, such as radium and beryllium, and depend on the scattering and slowing of fast neutrons by hydrogen.

Element analysis has been made of concrete materials by neutron activation methods, primarily silicon and calcium (1, 10, 16). The fast neutron activation of silicon-28 gives aluminum-28 that decays with a 2.3-min half-life yielding a 1.78-MeV gamma ray for measurement. The thermal neutron capture by calcium-48, which makes up 0.18 percent of natural calcium, gives calcium-49 that decays with an 8.8-min half-life yielding both 3.09 and 4.05 MeV photons. A portable positive ion accelerator (operating from a station wagon) provided 14.3 MeV neutrons for use with concrete pavement (1).

The possibility of concrete additives, such as manganese, that are easily activated has been considered as an analytical aid; tests were also done toward monitoring with the natural radioactivity in concrete materials (1). Unfortunately, neither of these schemes has proved particularly promising.

NUCLEAR SPECTROSCOPIC TECHNIQUES

Element identification by neutron absorption can be accomplished by the measurement of neutron-capture gamma rays or decay gamma rays. When a nucleus captures a neutron, gamma rays are always emitted immediately (prompt gamma radiation) in about 10^{-14} sec. In addition, the product nucleus may be radioactive and will emit additional gamma rays (delayed gamma radiation) while it undergoes decay to a stable state. The gamma energy spectrum of the emitted radiation, either prompt or delayed, is a characteristic signature of the isotopes of an element. In general, the delayed gamma spectrum of a single element is simpler in structure than the neutron-capture or prompt gamma spectrum. In addition, delayed gamma rays are usually less than 2 MeV, whereas capture gamma rays range as high as 10 MeV.

Most analytical applications of gamma ray spectroscopy in the past have been made with delayed or decay gamma rays. Quite often, interferences are reduced by the use of chemical separation to assist delayed gamma activation analysis. With bulk materials, the penetration of low-energy gamma rays may limit the volume of the analytical sample. Smaller sized samples may lead to lower sensitivity for highly disseminated elements of low concentration, and, if the minerals are nonuniformly distributed, the analyses of small samples may not be statistically significant. In addition, the natural background from the thorium, uranium, and potassium decay may complicate the delayed gamma radiation measurements.

Many elements do not respond readily to decay gamma ray analysis because the nuclides formed are not radioactive or do not have convenient half-lives, e.g., silicon, calcium, aluminum, and iron. In contrast, all elements yield neutron-capture gamma rays. Consequently, the potential application of this approach may be greater. Of course, in either decay or capture gamma ray methods, the neutron absorption rate must be high enough to yield a measurable spectra of the element of interest. For these reasons and as a result of the original studies with germanium detectors on pure elements and mixtures by El Kady (3) and Wiggins (4), our preliminary experiments with concrete materials have been made with the prompt gamma radiation techniques. Also, it was recognized that the generally higher energies of capture gamma rays may have advantages over the lower energies of decay gamma rays. These more energetic photons suffer less interaction in reaching the detector, and this may, as indicated above, allow the examination of larger samples of disseminated materials (and presumably with enhanced sensitivity). Moreover, the interference from any decay gamma rays is lessened by measurements of only the high-energy capture gamma rays.

GAMMA RAY DETECTORS

For any spectroscopic method, an appropriate radiation detector is needed; and, until recently, generally poor resolution of detectors made measurement of high-energy gamma rays difficult. With lithium drifted germanium, Ge(Li), detectors, the resolution is good enough so that it is now possible to identify readily high-energy capture gamma rays. In addition, the pair production (positron and electron) produced in the detector by the high-energy gamma rays can aid element identification. The annihilation of the positron produces two 0.51-MeV photons, one or both of which may escape from the Ge(Li) crystal; thus, for high-energy gamma rays, 3 spectral peaks result for each fundamental or full energy peak. These are designated the full peak, $E\gamma$, the single escape peak, $E\gamma$ - 0.51 MeV, and the double escape peak, $E\gamma$ - 1.02 MeV. The spectrum of iron as the oxide is shown in Figure 1 and illustrates the escape effect of the annihilation photons. (Energies on all curves are in MeV.) The dominant full-energy peak (f) of iron at 7.64 MeV is actually 2 closely spaced peaks (a doublet). The larger single-escape peak (s) at 7.13 MeV and the very large double-escape peak (d) at 6.62 MeV are also doublets. This spectrum shows the greater efficiencies of a larger Ge(Li) crystal (about 50 cm^3 active volume) for the high-energy escape peaks compared to the full peak. The multiplicity of peaks may complicate a spectrum, but, because a high-energy gamma ray is revealed 3 times, this trio of peaks assists in identifying an element that may be masked by the prompt gamma rays of other constituents.

ELEMENT SENSITIVITY

As indicated, the number of gamma rays produced in a nuclide depends on the neutron absorption that is reflected by the ratio of σ/A, where σ is the microscopic absorption cross section in barns (10^{-24} cm^2) and A is the mass in atomic mass units. Moreover, the gamma ray yields are equally important, and this is expressed by the number of gamma rays I of a particular energy emitted per 100 neutrons absorbed. The values of I are well known and have been measured and compiled (13, 14). The combination Iσ/A, then, gives a sensitivity index that has proved useful in predictions of applications of the technique.

The sensitivities, Iσ/A, for the high energies of interest have been tabulated (17). Lately, another tabulation for the low-energy capture gamma rays has been made (18). Spectral contrast is very important, for a high absorber with many gamma ray energies, e.g., cadmium, may give a bland spectrum, while iron, as shown in Figure 2, with a few prominent lines yields the better spectrum (17). (Iron proved useful in calibration.) The sensitivities and past experimental work (3, 4) show that for the higher energies Ti, Cr, Mn, Fe, Co, Ni, Cu, S, Cl, Mg, and Au respond well to analysis by capture gamma rays.

In addition, a number of elements that have a relatively low sensitivity but are often found in high concentration in materials of industrial interest appear suitable for this technique. Table 1 gives elements in concrete and the sensitivity values of the most interesting prompt photon lines for analytical studies. If the sensitivities and the concentrations of elements in concrete are considered, iron is expected to be most easily identified in basaltic concretes and calcium in those with limestone aggregates. For equivalent concentration, silicon should respond about as well as calcium. Aluminum, magnesium, and sulfur should give a detectable but lower signal.

NEUTRON SOURCES

Neutrons for analyzing bulk samples can be obtained from a nuclear reactor, an accelerator, or an isotopic source. Using a reactor requires transporting the sample to that facility, and most of the analysis with decay radiations has been made with reactors. Reactors with their safety, licensing, and scheduling problems may prove inconvenient for some applications. Accelerators, which are more likely to serve single users, offer portability and, as mentioned, have been applied to concrete analysis using a decay gamma ray method (1). Isotopic sources that depend on the alpha-neutron re-

Figure 1. Iron oxide partial-capture gamma ray spectrum, 10 min and 243 grams of Fe_2O_3.
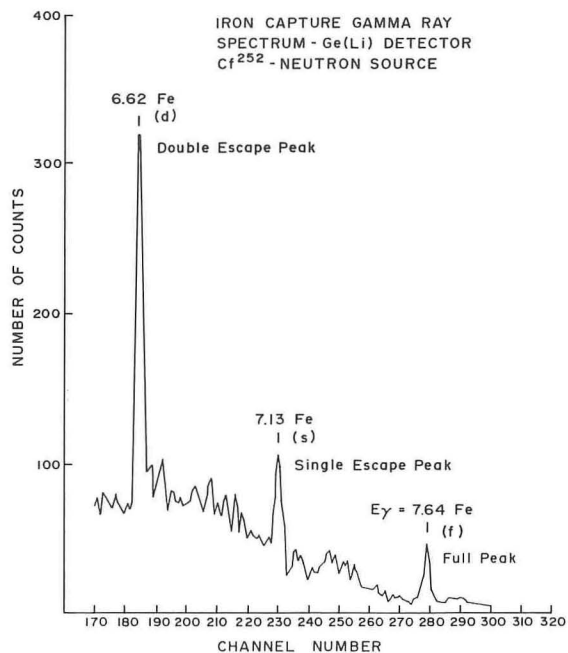
Figure 2. High energy neutron-capture gamma ray spectrum of iron, 100 min and 243 grams of Fe_2O_3.
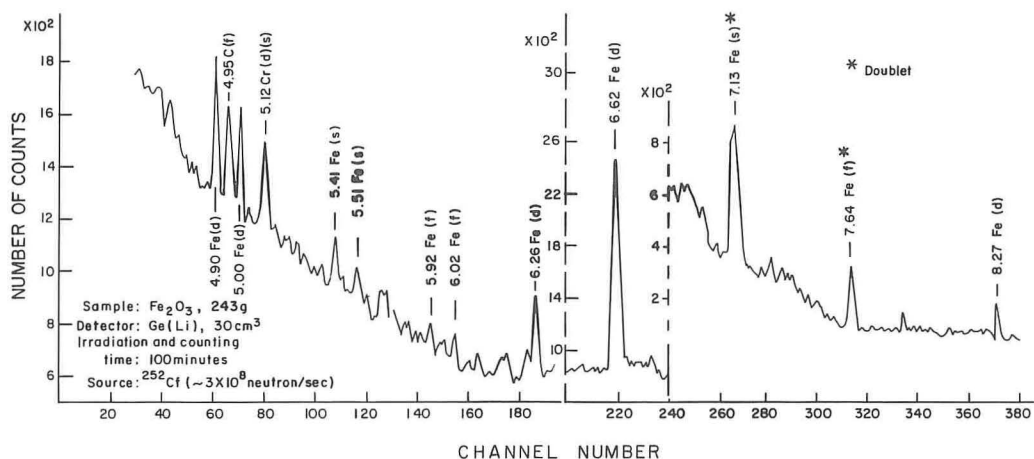
Table 1. Capture gamma ray sensitivities of elements in concrete.

Element	Cross Section (barn)	Atomic Mass Unit	Energy (MeV)	I[a]	Iσ/A
Mg	0.063	24.312	3.916	40.82	0.10
			3.054	10.83	0.028
Al	0.235	26.981	7.724	20.10	0.175
Si	0.160	28.09	6.380	12.62	0.071
			4.934	70.55	0.402
			3.539	79.58	0.453
S	0.512	32.064	5.420	42.44	0.678
			3.221	19.46	0.311
Ca	0.430	40.08	6.420	28.09	0.301
			4.419	10.79	0.116
Fe	2.62	55.847	7.646[b]	22.14	1.04
			7.632[b]	27.19	1.27
			6.018	8.08	0.379
			5.921	8.29	0.389

[a]Number of photons emitted per 100 neutrons absorbed.
[b]These Fe peaks are not resolved in Figures 1 and 2.

action, such as radium, polonium, or plutonium with beryllium, have been used for analytical studies, but the neutron yield is low; most applications with isotopic sources have been with decay radiation, but some work has been reported on the use of prompt gamma rays (19).

Recently, californium-252, a synthetic isotope that undergoes spontaneous fission with a half-life of 2.62 years, has provided a neutron source that can compete in yield with an accelerator and even a low-power reactor (20). For experiments such as those described in this research, the physical size of the californium source is small, and the cost, safety, and licensing problems are much less than those with a reactor. Also, because it is a sealed source, there is essentially no maintenance problem. The low cost and portability indicate that installations of californium-252 as a part of processing control arrangement or for field use should be feasible (15).

CALIFORNIUM SOURCE

The californium-252 source used for this work was approximately 100 μg, yielding about 3×10^8 neutrons/sec. The neutron energy distribution is similar to that from uranium-235 fission in nuclear reactors, but the average neutron energy for the californium-252 source is slightly higher. The associated gamma ray energies from fission and the fission products of californium-252 are generally lower than those of interest in this work. The californium was in the form of the oxide, Cf_2O_3, and was doubly encapsulated. The inner capsule was made of an alloy of 90 percent platinum and 10 percent rhodium. The Cf_2O_3 was held between 2 small, type 316 stainless steel frits within the capsule. The outer capsule was made of zircalloy-2. (A source encapsulated with stainless steel, type 304L, was used in early experiments but not for the measurements reported here.) These later construction materials were incorporated by the fabricator, the Savannah River Laboratory, to reduce the capture gamma ray background (11, 12). The source outside dimensions were 1.33 by 0.37 in. in diameter; it was held in the end of a 3-ft phenolic (paper base) rod $1\frac{1}{2}$-in. in diameter.

IRRADIATION ASSEMBLY

The irradiations were performed with this source in a plastic (polymethylmethacrylate) tank 26 by 22 by 26 in. high filled with water (Fig. 3). Cardboard boxes of paraffin were outside the tank and provided 10 in. of additional shielding. The californium source was located within the tank at one side near the bottom. The samples were placed in a given position outside of the tank near the source, as shown. Samples of the cement and granulated aggregate were irradiated in plastic dishes 6 in. in diameter and $\frac{3}{4}$ in. thick, and the concrete was in blocks, 3 by 4 by 16 in. The Ge(Li) crystal (cooled with liquid nitrogen and shielded with lead, boron as Boral, and cadmium) was positioned on the side of the tank opposite the sample. This provided considerable moderator (water and paraffin) and absorber to lower the outside gamma ray background and to protect the Ge(Li) crystal from neutron damage. A 400-channel analyzer, with a field effect transister (FET) preamplifier, a power supply, an amplifier, a biased amplifier, and a plotter served the detector system.

AGGREGATES, CEMENT, AND CONCRETE MIXTURES

Aggregates in common use but of diverse composition were chosen to demonstrate the procedure: quartzite sand from Branchville, Maryland; quartzite gravel from White Marsh, Maryland; limestone from State College, Pennsylvania; and basaltic rock, a diabase, from Chantilly, Virginia. The cement was a blend of type 1 brands. The composition of these raw materials is given in Table 2.

The concrete mixtures contained 5 or 7 sacks of cement/yd^3 and between 5 and 7 gal of water/sack of cement. The weight of sand aggregate was about 2 to 3 times the cement, and the coarse aggregate (gravel, limestone, or diabase) weight was about 20 to 50 percent more than the fine aggregates. These proportions were chosen more for experimental convenience rather than for quality of concrete. From this compounding, the compositions given in Table 3 on a weight basis for the 7 concrete batches were obtained by calculation.

RESULTS

The constituents that make up concrete, i.e., cement, sand, and coarse aggregates, were irradiated in the experimental arrangement shown in Figure 3. Figure 4 shows the prompt photon spectrum from the cement. The calcium peaks are at 5.91 MeV (single escape, s) and at 5.40 MeV (double escape, d). Silicon is observed at 4.93 MeV (full, f) and 4.42 MeV (s). Lines at 5.63 MeV (d) and 5.12 MeV (s) are from neutron-proton reaction in oxygen to produce the short-lived nitrogen-16. This oxygen was primarily in the water around the neutron source and, hence, appears in all spectra. The spectrum of the diabase rock sample (not shown) reveals mainly the relatively strong 6.62 MeV (d) iron line; also the less intense calcium and silicon lines from the feldspar minerals of the diabase are apparent. The response of the sand and gravel shows primarily the 4.93 MeV (f) and the 4.42 MeV (s) silicon peaks. Figure 5 shows the gamma ray response of the limestone aggregate. The 5.40 MeV (d) calcium line is the most prominent.

The neutron-capture gamma ray spectrum of a concrete block with a coarse gravel aggregate (batch 2) is shown in Figure 6. A relatively small amount of iron is indicated by the 6.62 MeV (d) line. Calcium in the cement is noted by the 5.40 MeV (d) peak, and the silicon lines from both sand and gravel at 4.93 (f) and 4.42 MeV (s) are clear. Figure 7 shows the response of a concrete block with a diabase aggregate (batch 5). The significant 6.62 MeV (d) iron peak and the calcium and silicon are readily apparent. Figure 8 shows the prompt gamma ray spectrum of the limestone aggregate concrete (batch 3) block. The relatively large calcium line from the aggregate and cement is at 5.40 MeV (d), and the silicon peaks at 4.93 (f) and 4.42 MeV (s) are from the sand. The weak peak at 6.62 MeV (d) is expected because of the small amount of iron present.

In general, the intensities of the prompt gamma ray peaks of the spectra reflect the relative composition given in Table 2. The sensitivity curves, shown in Figure 9 for Fe_2O_3, SiO_2, and CaO for the concrete blocks tested, are in general agreement with the percentages given in Table 3. The experimental results indicate that iron, calcium, and silicon can be determined in finished concrete and its feed materials with this kind of instrumentation and that iron is the easiest to determine. This is in accord with the sensitivities, $I\sigma/A$, and earlier experiments with pure materials and geologic samples. The aluminum is revealed by the 7.72 MeV line in the spectrum of the high aluminum materials, but at a lower intensity. Other studies lead us to believe that, with more recent electronic equipment, aluminum as well as sulfur can be seen. In addition, from these other studies, we are confident of being able to refine the accuracy limits.

It is realized that elemental content of concrete, in itself, may not be so important as other features, such as cement and water content. However, with a knowledge of the feed material composition and the composition of the finished concrete, this other important analytical information can be deduced.

SUGGESTED APPLICATION

The experiments were performed to show the feasibility of using the capture gamma ray technique in processing plants for concrete materials and in the field for the formed product. Of course, the technique could also be applied in a laboratory near the concrete operation. In either case, an analytical assembly of a shielded californium source and Ge(Li) detector with electronics would be needed. Analytical assemblies (15) have been proposed by the authors for process control, e.g., iron and copper; these could be adapted for concrete. Obtaining the scan of the entire spectrum would not be required; instead only certain energy lines need be counted. This information could be used in turn to control the operation, such as material flow in a process system. For field work, portability is required, and perhaps a mobile laboratory would be useful. Electrical power and liquid nitrogen would be required (a 30-liter supply of nitrogen has been sufficient for the detector for 2 weeks).

For formed concrete, an analytical assembly could be used on roads, foundations, and other reinforced concrete structures (reinforcing iron should be readily identifiable,

Figure 3. Experimental arrangement.
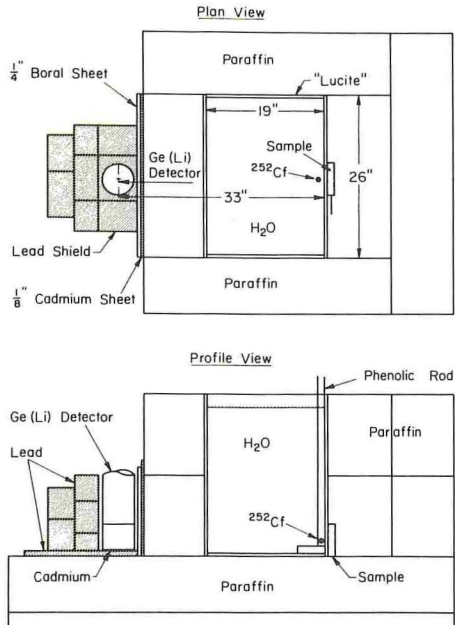
Table 2. Percentage composition of concrete aggregates.

Compound	Cement	Sand	Gravel	Limestone	Diabase
SiO_2	21	97	98	4	53
CaO	63	—	—	52	9
Al_2O_3	5	2	1	—	17
Fe_2O_3	3	0.8	0.5	—	14
MgO	3	—	—	1	4
SO_3	3	—	—	—	0.3
CO_2	—	—	—	42	—[a]

[a]Also some Na_2O, TiO_2, and MnO_2.

Table 3. Composition of concrete batches.

Compound	Gravel Batch 1	Gravel Batch 2	Lime-stone Batch 3	Lime-stone Batch 4	Diabase Batch 5	Diabase and Gravel Batch 6	Gravel Batch 7
Content (percent)							
CaO	7.5	11	30	34	14	13	11
SiO_2	81	78	40	36	60	68	77
Al_2O_3	1.8	1.9	1.3	1.4	9	6	2
Fe_2O_3	0.9	1	0.6	0.7	7	4	1
MgO	0.4	0.5	0.7	0.9	2	1	0.5
SO_3	0.4	0.5	0.4	0.5	0.5	0.5	0.5
CO_2	—	—	18	18	—	—	—
H_2O	8	8	9	9	7	7	8
Density (lb/ft^3)							
—	144	144	147	147	147	145	139

Note: Batches 1 and 3 were with 5 sacks of cement per cubic yard concrete, and the others were with 7 sacks.

Figure 4. Neutron-capture gamma ray spectrum of cement (380.5 grams).
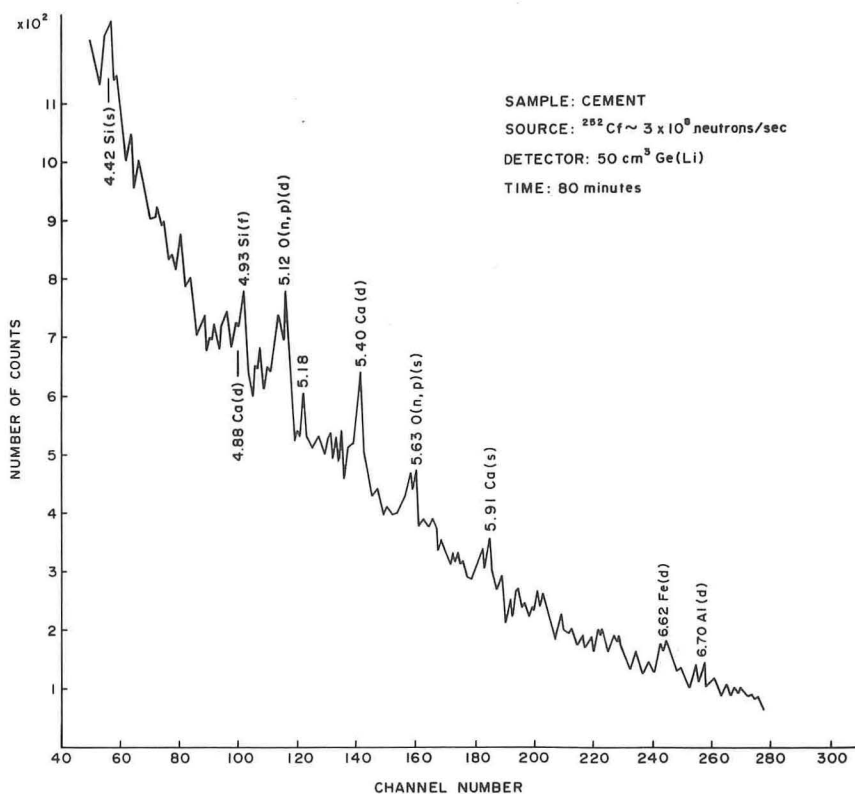

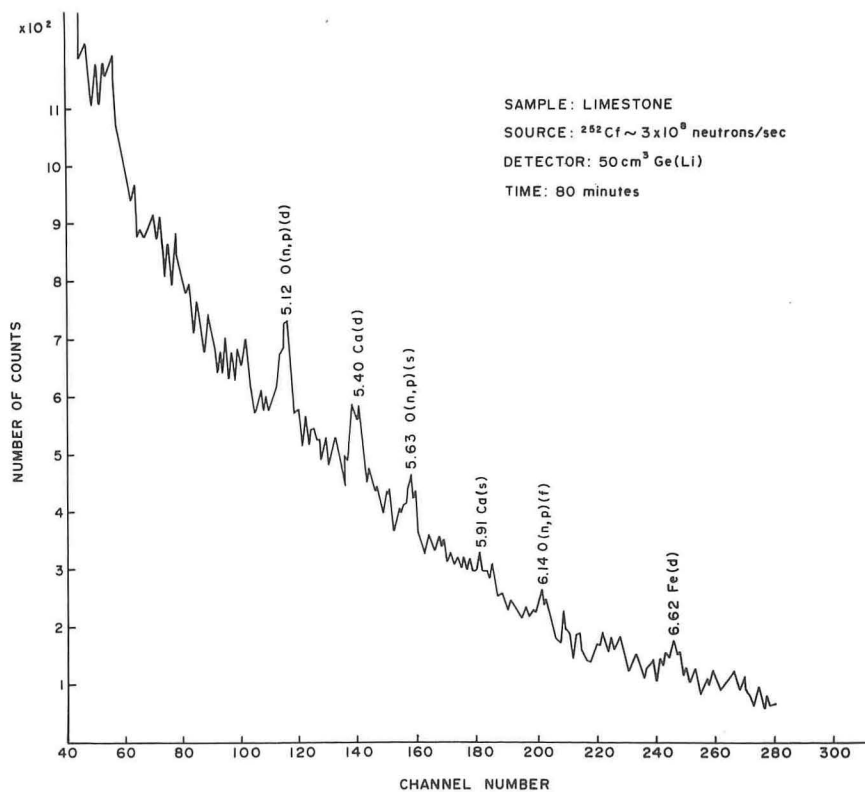**Figure 5.** Neutron-capture gamma ray spectrum of limestone (519 grams).

Figure 6. Neutron-capture gamma ray spectrum of concrete with gravel aggregate.
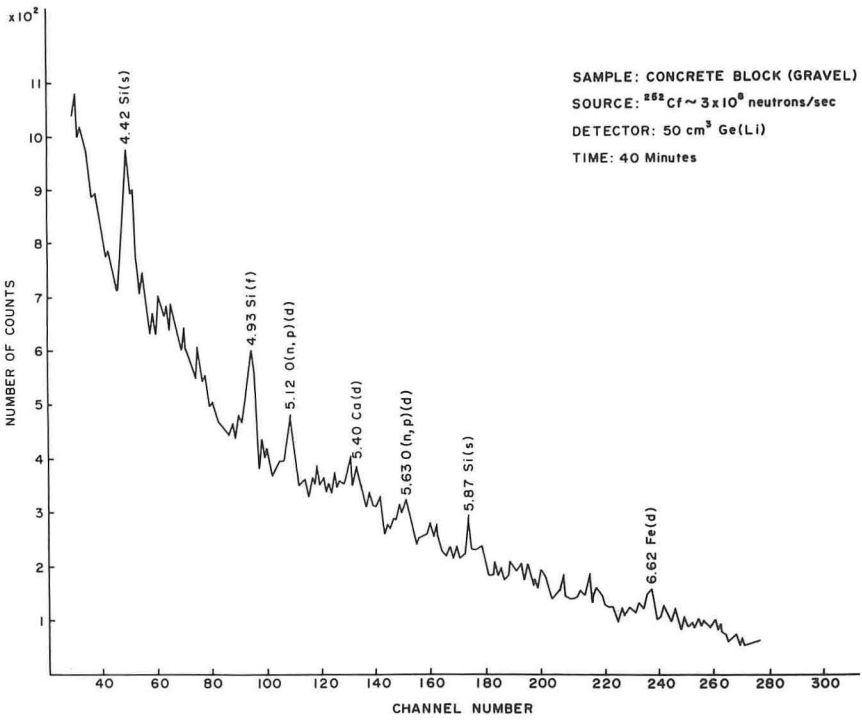

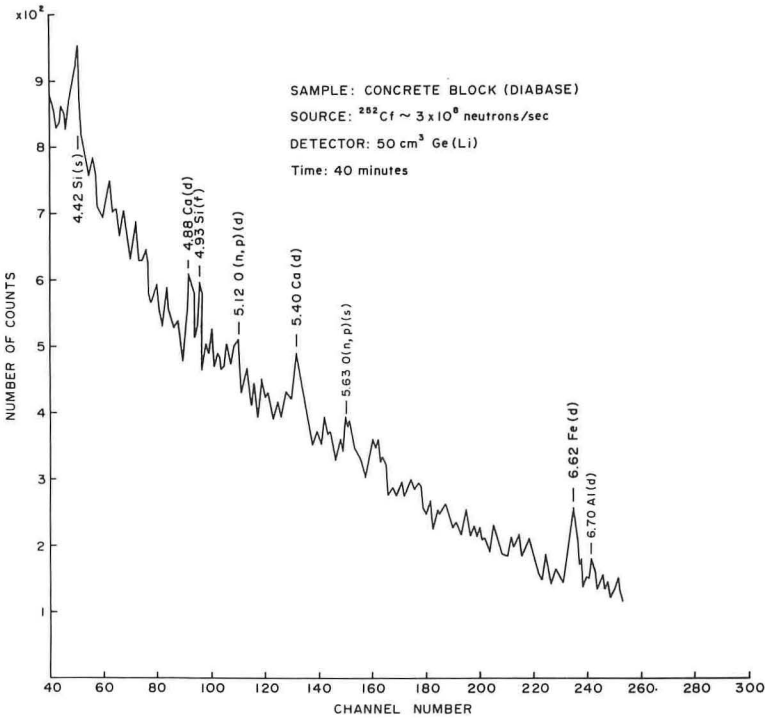**Figure 7. Neutron-capture gamma ray spectrum of concrete with diabase aggregate.**

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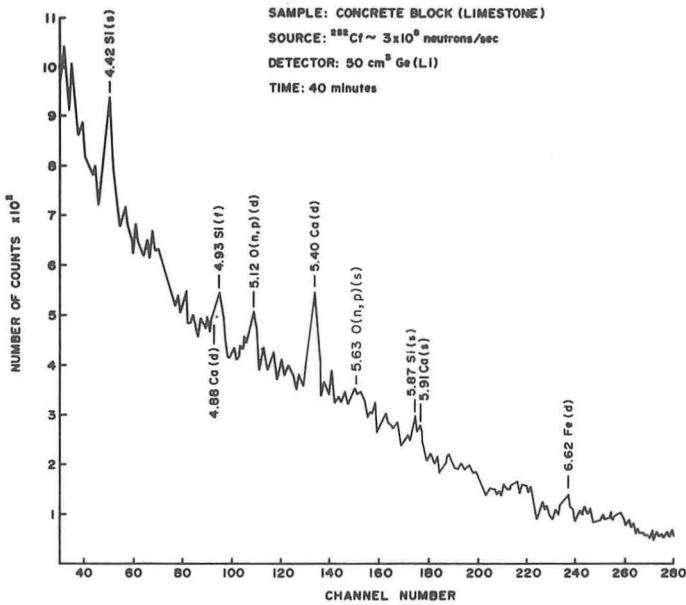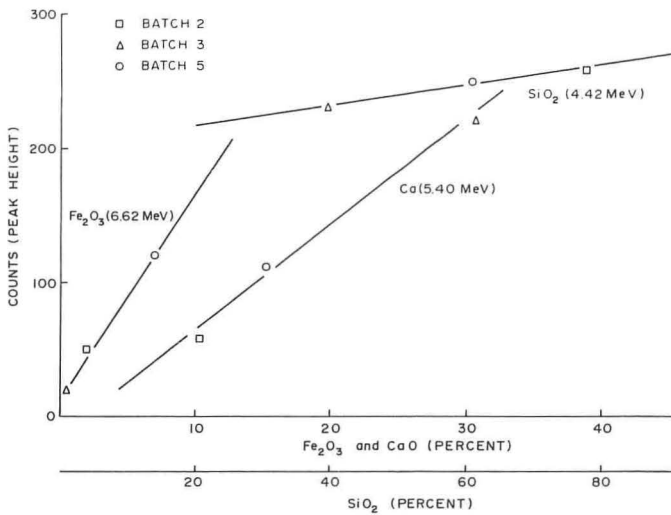Figure 8. Neutron-capture gamma ray spectrum of concrete with limestone aggregate.

Figure 9. Sensitivity curves for concrete block.

although concrete inspection procedures should, presumably, have already revealed this). As indicated, concrete with the more basic rock aggregates, e.g., traprock of Washington, Oregon, Connecticut, New Jersey, and northern Virginia, should be particularly suitable because of the high iron content.

Monitoring the concrete-mixing process may be an application if the installation is large. The dry raw materials in bins or on conveyors and the wet mixture could be analyzed. However, with the smaller batch plants, the amounts of materials are easily weighed, and the component compositions are generally uniform and are well known; consequently, nuclear analysis does not appear to offer advantages for small operations. Use of the technique for the aggregate preparation processes should be possible; but, as with the smaller plants, there does not appear to be the incentive for such specialized control.

Monitoring the cement process itself by nuclear spectroscopy might be attractive. Here the tonnage is high, and the slow kiln process with sizable inventories necessitates careful control of the streams to ensure a satisfactory cement. The process streams may be in bins and on belts or in water or in air. Monitoring all these streams, as well as the waste streams, to meet environmental restrictions, e.g., for calcium, should be possible by this nuclear method. Checking the gypsum addition by sulfur analysis should also be possible. Concrete involving slag from steel plants might be analyzed for the slag iron and manganese.

In summary, this nuclear spectroscopic scheme appears to offer a new way to determine the components of concrete in situ with almost immediate information. The signals obtained allow process control and the possibility of integrated response and, hence, can yield information on inventories and production.

ACKNOWLEDGMENTS

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WATER CONTENT OF FRESH CONCRETE MEASURED BY NEUTRON RADIATION

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Experiments were made to study the use of neutron radiation to measure the water content of fresh concrete. Hydrogen in the water causes neutrons to be reduced in energy and backscattered to a detector that counts low-energy neutrons only. Thus, with proper control, the neutron count is proportional to water content in a standardized sample of concrete. Standard $^1/_2$-ft^3 concrete measures were modified to receive the nuclear probe (a tube carrying source and detector) and contained the concrete sample. The apparatus was calibrated with laboratory-made concretes having a wide range of water contents. Water content of concrete samples taken from truck-mixed batches measured between 0.92 and 1.11 times the average batched unit quantity of water. Readings taken with the probe located in different positions relative to polyethylene models of the sample showed a large influence of the spatial relation of source, detector, and sample. These tests demonstrated the applicability of the nuclear method for measuring water content of fresh concrete but indicate that refinements are needed to increase the precision obtainable.

•THE USE of neutron radiation to measure moisture content of soils and other materials is well established, and commerically available apparatus for this purpose is convenient to use, rugged, and reliable. Experiments were made with such instruments to determine the feasibility of measuring directly and promptly the water content of freshly mixed portland cement concrete. In this procedure, a sample of freshly mixed portland cement concrete is irradiated with high-energy neutrons, and a portion of these are thermalized (reduced to a low-energy state) by collision with the hydrogen nuclei in the water. The resulting radioactive backscatter is measured and with proper calibration is an indicator of water content of the tested sample.

The determination of the water content of fresh concrete can be useful in controlling its quality and in providing assurance that it will be of the desired strength before it is placed. The slump test, although quick and direct, is not adequate for measuring water content or water-cement ratio because it is affected by factors other than water. The Dunagan (1) and Willis-Hime (2, 3) procedures for measuring the constituents of fresh concrete and the oven drying of samples to measure water content (4) have certain virtues, but they all require a relatively long time to complete after sampling. The use of backscattered thermal neutrons to measure water content of soils (5, 6, 7), asphalt content (8), and water content of concrete (9) is described elsewhere. Further experiments on concrete are described here.

FACTORS AFFECTING NEUTRON COUNTS

The radioactive source produces high-energy neutrons that collide with the nuclei of the elements in the sample surrounding the source. Three kinds of interaction take place (11). One is a reduction in energy by successive collisions with hydrogen nuclei, the process called thermalization, and at any time a certain proportion of the total

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available neutrons will be thermal neutrons at the lowest energy state of 0.025 eV. The second interaction is reflection from collision with heavier nuclei, which causes high-energy neutrons to rebound with essentially no loss in energy and to be moving in all directions. However, many neutrons escape collision and reflection and, hence, cannot interact in other ways. Third, neutrons can be absorbed by reaction with other nuclei, producing isotopes and other radiation; such reactions depend on the energy state of the neutron. Thus, the number of neutrons available to react with hydrogen and become thermal is the number emitted by the source less the number that escape or react with other nuclei. Of the number of neutrons available, the number that will become thermal depends on the amount of hydrogen. Hence, a count of thermal neutrons, taken under controlled conditions, is an indication of the amount of hydrogen present in the sample.

Thermal neutrons are counted by detectors that consist of tubular elements containing boron trifluoride gas, enriched in the boron-10 isotope. A high electrical potential (1,400 to 1,500 volts) is maintained between electrodes in the gas. When a thermal neutron strikes the boron-10 nucleus of the boron trifluoride molecule, an alpha particle is ejected. The electrically charged alpha particle is highly ionizing in the boron trifluoride gas, causing a pulse between the electrodes. This pulse is amplified and triggers an electronic counter capable of summing all such pulses that occur within an accurately measured interval of time.

Several factors, as well as the amount of hydrogen present, affect the number of thermal neutrons counted. The intensity of the source, of course, governs the total number of neutrons available in a given time. Because neutron emission is not steady but rather occurs in bursts, a sufficiently long time is needed to get an essentially constant emission in the counting time interval. The size and shape of the detectors, their distance from the source, and their orientation to the source affect the proportion of thermal neutrons that will be intercepted by the detectors and hence counted. It is essential that the electrical potential in the detectors and the amplifier gain remain constant so that a calibration made under one set of these parameters will be applicable during use of the apparatus. Obviously, the electrical circuitry must be adequately shielded against external events that could produce pulses to activate the counter.

If the volume of material surrounding source and detector is large enough, the thermal neutron count will depend on the total amount of hydrogen within the volume affected by the radiation. The size of this volume, in which detectable thermalization occurs, increases as the hydrogen atom concentration decreases. On the other hand, a sample of smaller size and shape will permit a larger share of high-energy neutrons to escape, and, hence, the count of backscattered thermal neutrons will be smaller than would be obtained from a larger sample having the same concentration of hydrogen per unit volume. Thus, the size and shape of the sample and the positions of source and detector tubes relative to the sample must be held fixed to provide an accurate calibration. It is important also that there be no water or other hydrogen-containing material outside the sample but within the volume of detectable thermalization.

The composition of the medium may contribute to the thermal neutron count in addition to the count arising from the hydrogen atoms in the water. If hydrogen is present in any form, for example, in organic matter or hydroxides, it will be counted the same as hydrogen in water. Thermalization caused by other lightweight elements will be detected as well. On the other hand, certain elements that may be present cause a reduction in count inasmuch as they are capable of reacting with and absorbing neutrons. Neutrons so absorbed are not available to be thermalized and counted. Heavy elements present in the material reflect neutrons, preventing their escape and increasing the proportion of those emitted that are available for thermalization and detection. These potential sources of error in measuring water content can be eliminated if counts are taken on the dry ingredients of the concrete. Background counts determined on dry materials are subtracted from the counts obtained on concrete samples to give the net count indicative of water content.

APPARATUS AND PROCEDURE

Two pieces of apparatus were used in these tests. This equipment was commercially available and was designed for moisture measurement in soil at depths below the surface.

Each consisted of 3 parts—probe, shield, and scaler—with electrical cable connecting probe and scaler. The probe consisted of a closed metal tube a little more than 1 in. in diameter and about 1 ft long that contained the neutron source, detector, and amplifier. The lower end of the probe contained shielding to prevent the escape of radiation from the bottom end of the probe. When not in use to test a sample, the probe was housed in the shield to protect the operator from radiation. The scaler provided the electrical power to charge the detector and operate the amplifier and pulse counter. The counters were provided with automatic timing switches to limit the counting time interval.

The neutron source in probe A was 100 millicuries of americium-beryllium, which emitted fast neutrons at a rate of 2.08×10^5/sec. The source in probe B was a 4.09-millicurie source of radium-beryllium that emitted 4.91×10^4 fast neutrons/sec. Boron trifluoride detectors were used in both probes.

The experimental procedures adopted were designed to be used without modification to the commercially available apparatus. This required that the source and detector in the probe be located within the concrete test sample. It was also felt desirable to transfer the source in the probe from its shield to the test position within the sample without exposing the operator to direct radiation. The apparatus used is shown in Figure 1. It consists of a modified unit weight measure containing the concrete sample with a central access tube in which the probe was placed. The shield was supported above the concrete by an extension sleeve on the access tube. The scaler was located on a nearby bench.

Unit weight measures of 1-ft^3 and $\frac{1}{2}$-ft^3 capacities were provided with a central steel tube welded to the reinforced base. This tube was just large enough to accommodate the probe, keeping it free from contact with the concrete. This prevented wear on the probe and possible jarring of the electronic elements that might occur if the probe were in contact with the concrete. It also eliminated the need to clean the probe and protected the operator from unnecessary exposure to radiation. The tube was cut off $^3/_4$-in. below the level of the top of the measure and provided with a cap so that the top of the tube would not interfere with filling, compacting, and striking off the measure. The volumes of the modified measures were calibrated so that accurate unit weight values could be calculated.

Preliminary tests were made to ascertain shielding requirements, optimum operating voltages for the probes, standard count readings, variation in count rate with length of counting time, and background counts in air and in empty containers. Radiation safety surveys showed that no special shielding was needed. The standard count was taken with the probe in a fixed position relative to its shield so that comparative readings could be taken from day to day with the probe under exactly the same conditions. This provided a check on the operation of the entire system—source, probe, and scaler. A malfunction in the operation of the equipment would, therefore, show up as a change in the standard count. Background counts taken with the probe in the air and in the empty containers showed such low readings that no significant errors occurred from this source (9).

The procedure followed in taking nuclear readings was simple and straightforward. The sample material was prepared, and the measure was filled, compacted, leveled off, and weighed. The access tube was then uncapped, the tube extension sleeve was affixed, and a plywood cover was placed over the measure to minimize evaporation. The probe in its shield was placed in position over the measure, and the probe was lowered into the access tube. The operator than walked away from the unit, started the count on the scaler, and remained several feet away until the count was complete. The count and its elapsed time were recorded, the probe was drawn up into the shield, and the assembly was returned to its case until needed for the next observation. Standard counts, with the probe in its shield, were taken at the beginning and at the end of each period of work in the laboratory.

Samples of cement and oven-dried sand gravel were tested to determine whether these ingredients of the concrete would affect the neutron counts. The measures were filled with each of these materials in turn, and counts were taken. The thermal neutron counts were so low that no corrections were necessary for thermalizing elements in the dry constituents of the concrete in these experiments.

CALIBRATION OF PROBES

The probes were calibrated with concrete and mortar mixes covering a wide range in water contents. This procedure gave a direct calibration on concrete materials and also provided data to judge the accuracy and reliability of the calibrations. Water content varied from about 9 to 15 lb/ft^3 (30 to 50 gal/yd^3) although usual concrete mixes are found in the lower end (30 to 40 gal/yd^3) of this range. However, when the largest range was used that could be obtained without undue segregation of the mixtures, the calibration could be established more definitely.

The results of the calibrations for water content are shown in Figures 2 and 3. The abscissas are total water content, including absorbed water, and the ordinates are thermal neutron count ratio. The quantity of water in the measure was found by proportion of weights from the known quantity of water batched, the total batch weight, and the sample weight. Weight of water per cubic foot was calculated from the calibrated volumes of the measures and converted to units of gallons per cubic yard. Neutron counts are expressed in terms of the thermal neutron count ratio, N_R, which is the ratio of the test count divided by the standard count for the same time interval. Each plotted point represents a test batch of concrete. Linear regression curves were fitted to the data by the method of least squares, and these lines together with their equations are shown in the figures. The dashed lines are the limits of the 95 percent confidence interval. It is 95 percent certain that the actual water content of a tested sample as determined by this procedure and on these materials lies within the limits of the confidence interval shown. The index of correlation, I, for each curve shows the per-unit proportion of the data used for the curve that are within 1 standard deviation from the curve. The somewhat greater spread of the data points at higher water contents is probably caused by segregation of aggregates or bleeding in these wetter mixes.

These calibration curves show the effect of the sample size. Neutron counts taken on $\frac{1}{2}$-ft^3 samples were considerably less than those taken on the 1-ft^3 samples. Within the range of water contents covered, a much larger proportion of the neutrons emitted by the source escaped thermalization and detection when the smaller sample was used.

TRUCK-MIXED TESTS

A field study of the homogeneity of ready-mixed concrete (10) offered an opportunity to try the neutron measurement of water content against carefully controlled full-sized, truck-mixed batches of fresh concrete. This study was conducted by the engineering staff of the National Ready-Mixed Concrete Association to ascertain the effects of size of batch, mixing revolutions, mixing speed, and loading methods on the homogeneity of concrete within a batch. Unit weight, air content, slump, coarse aggregate content, and compressive strength tests were made on samples taken at intervals of about 15, 50, and 85 percent of the discharge of each batch. Homogeneity of the batch was judged by the closeness of agreement between the test results for the 3 samples. In these tests, truck mixers were charged with loads ranging from 7 to 9 yd^3. Different methods of charging were used, and the factors under study were varied. The quantities of materials charged were measured accurately on new equipment in a batching plant constructed for this test program. Some batches showed a high degree of homogeneity; others failed in one respect or another to meet the ASTM C 94 criteria.

Water content was measured by thermal neutron count on samples of concrete taken from 46 truck-mixed batches in this program. A sample of about 2 ft^3 was taken after 85 percent of the mixer load was discharged. Counts were taken on $\frac{1}{2}$ ft^3 of this sample; probe B was used, and the same procedure as described above was followed for calibration of the probe.

The results of these experiments are given in Table 1. Each entry in the table is the test for 1 truck-mixed batch. The results are presented in ascending order of batched water content. It is not the order in which the tests were made, nor were 2 batches at the same water content loaded into the mixer or mixed in the same way.

The batched water content was calculated from the total weights of materials loaded into the mixer, with correction for the air content. The measured water content was

Figure 1. Arrangement of apparatus for measuring radiation in concrete samples.

CAP
SAMPLE
ACCESS TUBE
MEASURE

a. FILLED MEASURE

SHIELD
CABLE
COVER
ACCESS TUBE EXTENSION
AMPLIFIER
DETECTOR
SCALER
PROBE ASSEMBLY
SOURCE
SHIELDING

b. COUNTING RADIATION
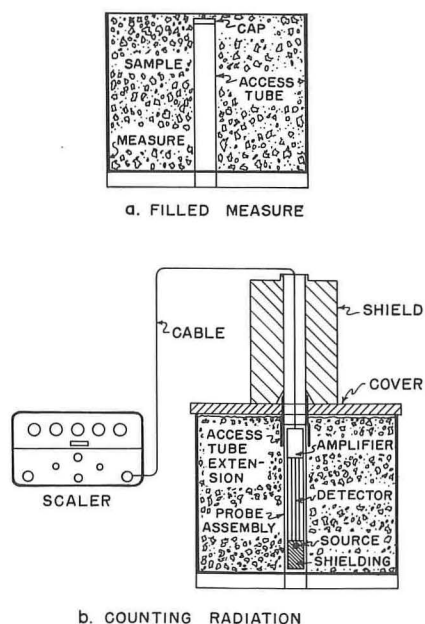
Figure 2. Thermal neutron count ratio versus water content, probe A.

1 CU. FT. MEASURE
$W = 1.10 + 120.5 \; N_R$
$I = 0.935$

COUNT RATIO, N_R

1/2 CU. FT. MEASURE
$W = 7.71 + 175.4 \; N_R$
$I = 0.918$

WATER CONTENT
GAL./CU. YD.
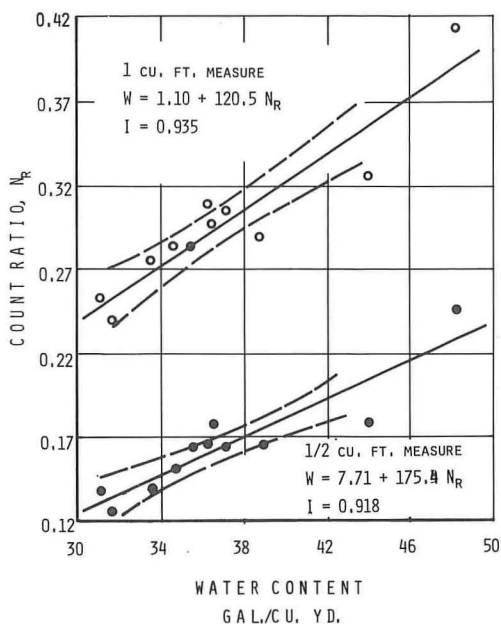
Figure 3. Thermal neutron count ratio versus water content, probe B.

1 CU. FT. MEASURE
$W = 8.0 + 28.6 \; N_R$
$I = 0.954$

COUNT RATIO, N_R

1/2 CU. FT. MEASURE
$W = 11.3 + 44.7 \; N_R$
$I = 0.941$

WATER CONTENT
GAL./CU. YD.
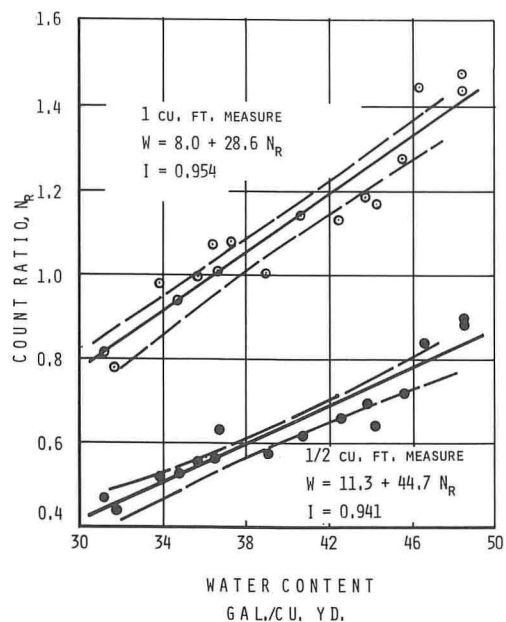
Table 1. Batched and measured water content of truck-mixed concrete.

Gal/Yd³			Gal/Yd³		
Batched	Measured	Ratio	Batched	Measured	Ratio
32.7	34.8	1.07	35.6	34.5	0.97
33.0	34.8	1.06		34.6	0.97
33.3	35.3	1.06		35.7	1.00
	36.6	1.10		37.3	1.04
	36.9	1.11	36.0	34.3	0.95
33.7	35.3	1.05		34.6	0.96
	35.4	1.05		35.2	0.98
	36.3	1.08		35.3	0.98
	36.3	1.08		35.8	0.99
	36.8	1.09		36.7	1.02
34.0	35.3	1.04		37.8	1.05
34.3	33.2	0.97	36.2	34.3	0.95
	35.8	1.04		35.2	0.97
	36.0	1.05		36.7	1.01
	36.0	1.05	36.5	33.8	0.92
	37.4	1.09		33.8	0.92
35.0	35.2	1.01		35.0	0.96
35.3	35.2	1.00		35.9	0.98
	35.4	1.00	36.9	33.9	0.92
	36.0	1.02		34.4	0.93
	37.4	1.06		34.6	0.94
	37.4	1.06		34.7	0.94
	38.9	1.10		36.6	0.99

Note: Average value of M/B ratio, 1.019; standard deviation in M/B ratio, 0.055, equivalent to 1.9 gal/yd³ water content.

determined from neutron counts taken with probe B and $\frac{1}{2}$-ft^3 samples, and the calibration data shown in Figure 3 were used. For comparison, the ratio of measured water content to batched water content is shown.

The correspondence between measured and batched water contents was not so good as had been hoped prior to the tests. Discrepancies as large as 10 percent were greater than one would want in a useful quality control procedure for fresh concrete. However, there are a number of potential sources of error in the procedure used here, as are discussed later, and these data were sufficiently encouraging to indicate the desirability of refining the method and apparatus to give more accurate measurements.

Another test of the validity of the water content measurements was made on some of the samples from the truck-mixed tests. Out of the total number of batches on which neutron counts were made, 6 showed a high degree of uniformity as judged by the other tests (such as slump and strength) that were performed. This was an indication that the water and cement were distributed uniformly throughout the batch; hence, the water-cement ratio found from the samples tested was a valid measure for the batch. Samples taken after about 15, 50, and 85 percent of the discharge were tested for water content by neutron measurement. Three 6- by 12-in. cylinders for compressive strength tests at 7 days were made from each of these 3 samples.

The results of this comparison are shown in the plot of compressive strength versus water-cement ratio in Figure 4. Four sets of points are shown. The crosses are plots of the average compressive strength for all cylinders of the batch against water-cement ratio as calculated from the weights of materials batched. The solid circles represent the average strength of the 3 cylinders made from the sample at 15 percent of discharge plotted against the water-cement ratio calculated from the average cement content and the water content found by neutron measurement on this sample. Similarly, the open circles and triangles represent these determinations for samples at 50 and 85 percent of discharge respectively. Complete readings were not taken on 2 batches; hence only the observed points are shown.

These data showed the results of the neutron-measured values to be consistent with the average strength versus water-cement ratio results for all batches. The range and average in water-cement ratios were a little higher for the individual samples than for the calculated batch averages. This may indeed be a correct indication of a small lack of homogeneity in these batches.

An attempt was made to measure water content directly on the concrete discharged into a 2-wheel buggy. A closed-bottom steel tube to accommodate the probe was welded to a 14-in. square steel plate. The tube was forced into the concrete in the buggy until the plate rested on the surface of the concrete with no air or free water trapped under it. The probe and shield were then placed on the plate, and the probe was lowered into the tube in the concrete. The arrangement was geometrically similar to the relation between probe, shield, and samples that pertained in the unit weight measures; the source was located at the same distance below the surface of the concrete in both cases. A separate sample of concrete was taken at the same point of discharge from the mixer as was each concrete buggy sample. This sample was compacted in the $\frac{1}{2}$-ft^3 measure, and neutron count readings were taken for comparison.

Thirteen batches were tested in this way for both the buggy and $\frac{1}{2}$-ft^3 samples; probe B was used for all. The ratio of count in buggy to count in $\frac{1}{2}$-ft^3 measure in these cases ranged from 2.04 to 2.67, and the other values were rather uniformly distributed between these extremes. The readings taken in the buggy were more than twice those in the $\frac{1}{2}$-ft^3 measure because the buggy contained a much larger volume of concrete to act on the neutrons. The variation in the ratio of count in buggy to count in measure was disappointingly large. Two causes of the variation were possible: segregation in the concrete in the buggy and, more important, the fact that the buggy was not filled to the same depth each time. This indicated that the size of sample in the buggy was not large enough to contain all of the zone of detectable thermalization around the probe. Hence, if measurements of water content are to be attempted in such vehicles or containers, it is necessary to standardize test conditions and determine a calibration for them.

COMPARISON BETWEEN PROBES

Measurements were taken with both instruments on the same sample or on samples of the same concrete in many of the laboratory and field experiments. The results of these observations are shown in Figure 5, where the readings (thermal neutron count/min) of probe B are the abscissas and those of probe A are the ordinates. Some points on this curve represent data taken on polyethylene layers in the container, as described in the following section. The agreement between the 2 instruments is very good for the entire range of water contents in both the laboratory and field tests. This is to be expected because the geometric relations between source, detector, and sample are similar for both probes.

EFFECTS OF SAMPLE GEOMETRY

An effort was made to study some of the variations observed in measurements on concrete. Tests were conducted on probe A by using polyethylene as the hydrogen-bearing medium in the $\frac{1}{2}$-ft^3 measure. The polyethylene was in the form of disks $\frac{1}{2}$ in. thick, with an inner 2-in. diameter hole to fit around the central tube in the measure and with an outer diameter $\frac{1}{4}$ in. less than the inner diameter of the measure. Different numbers of disks were arranged in the measure in several different spacings, and neutron counts were taken with the probe at several elevations in the measure.

A comparison between polyethylene and water is shown in Figure 6. One pound of water contains the same amount of hydrogen as 0.777 lb of polyethylene. Check readings were made by partly filling the measure with 21.3 lb of water, coming to a depth of 8 in., for which the neutron count ratio was 1.15. Then, 14 polyethylene disks, weighing 16.64 lb (an equal weight of hydrogen) and being 7 in. deep in the measure, gave a neutron count ratio of 1.12. This slight difference is caused by the difference of 1 in. to which the detector tubes were covered by the thermalizing medium.

Readings taken with different amounts of polyethylene in the form of 7, 11, and 14 disks uniformly spaced through the depth of the measure are shown in Figure 7. Neutron count ratios are plotted for each case as abscissas. The ordinates of the diagram are the locations of the radioactive source as the probe was raised by $\frac{1}{4}$-in. increments. With 7 disks, the neutron count ratios vary from 0.23 with the source $1\frac{5}{8}$ in. above the bottom of the measure to about 0.20 with the source $4\frac{5}{8}$ in. above the bottom of the measure. Larger ratios were found for the other cases with more polyethylene in the measure. The relative magnitudes of the count ratios for the same probe position are not linearly proportional to the weight of polyethylene in the measure. The fact that the count ratios increased disproportionately with increased weight of polyethylene indicates that the greater spacing between disks allowed more neutrons to escape when fewer disks were used.

The results of the third set of measurements using polyethylene are shown in Figure 8. In these experiments, a 4-in. thick layer (of 8 disks) of polyethylene was located at each of 3 positions in the measure: bottom, middle, and top. For each position of the thermalizing medium, the probe was raised so that the source moved through the full height of the measure. Neutron counts were taken at many different heights of source to give data for the 3 curves plotted.

With the polyethylene on the bottom of the measure, the neutron count ratio varied from about 0.50 with the source in its lowest position to a very small value as the probe was raised to midheight of the measure, and then to an increased value as more of the detector in the probe became encased in the shield. With the polyethylene in the middle position, the ratio increased as the source entered into the polyethylene, decreased as the amount of polyethylene between source and detectors decreased, then increased again as more of the probe was shielded.

In all of these tests, the detector tube entered the shielding as the probe was raised in the measure. Consequently, high-energy neutrons were thermalized within the shielding and were counted by the part of the detector tube within the shield.

Figure 4. Compressive strength for observed water-cement ratios, truck-mixed samples.

X Batch Average
● 15% Sample
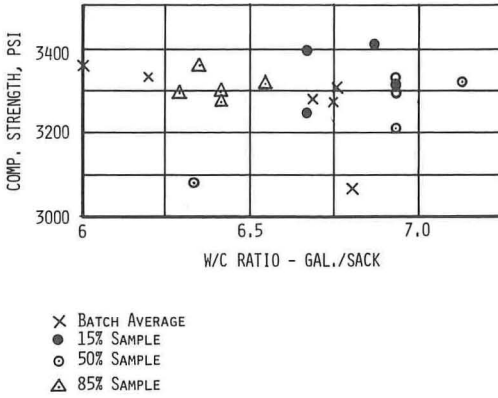⊙ 50% Sample
△ 85% Sample

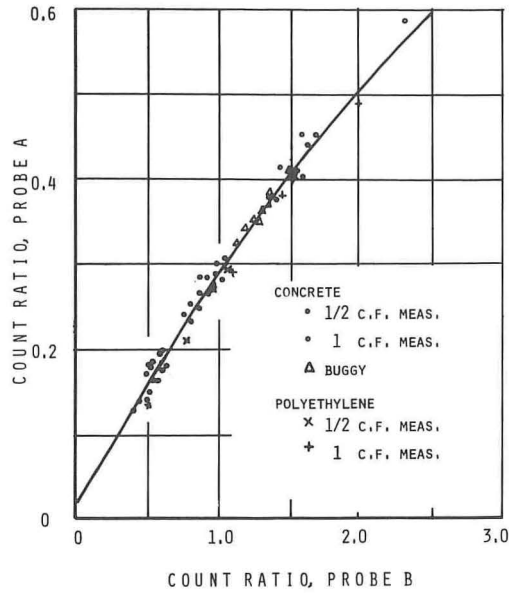## Figure 5. Comparison of probes.

Figure 6. Comparison of count ratios for equal weights of hydrogen.
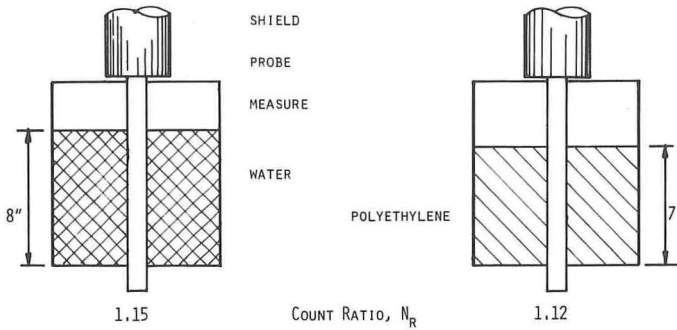
Figure 7. Effect of position of source on count ratio.

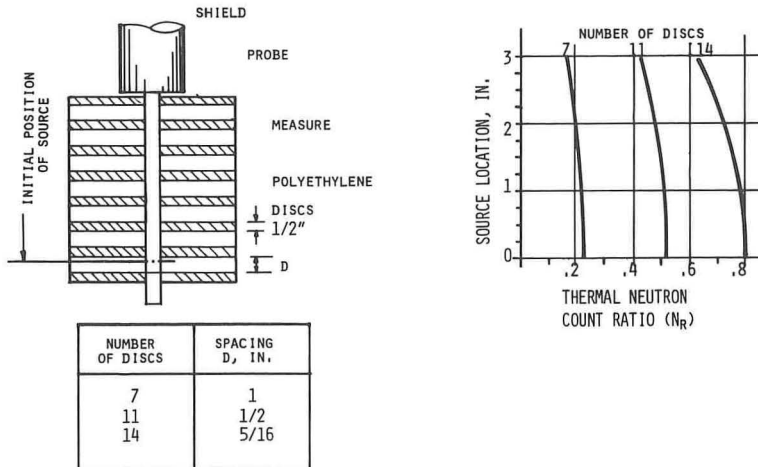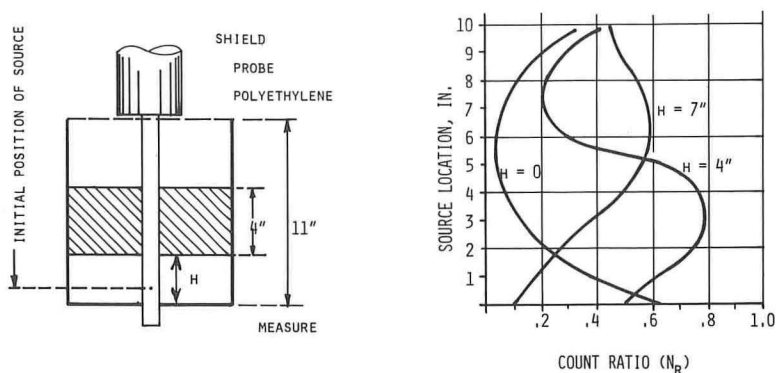| NUMBER OF DISCS	SPACING D, IN.
7	1
11	1/2
14	5/16

Figure 8. Effect of position of thermalizing material on count ratio.

DISCUSSION OF RESULTS

The results obtained thus far show that water content can be measured by this procedure of neutron irradiation and counting of the thermalized neutrons in a standardized sample. However, the results are not so precise as desired in a quality control procedure for concrete, and it is necessary to examine the possible sources of error or uncertainty in the procedure. It was expedient to use existing readily available equipment in these experiments to explore the feasibility of the idea. Changes in technique, apparatus, or test conditions might then be made to improve the precision of the results.

The size of the concrete samples must be larger than any used here to eliminate the size effect from the calibration. This would be impractical; hence, a standardized size and shape of concrete sample are essential.

Certain aspects of the problem of securing a representative sample of concrete have particularly significant effects on this method for finding water content. Variations in the composition of the sample, as caused by aggregate segregation or by bleeding, can cause error because the water content of the material nearest the source is most effective in the thermalizing process. Further, because mortar always will be closest to the tube containing the probe, the water content measured may be somewhat larger than the average for the batch. Aggregate size, although not studied experimentally here, will undoubtedly have an influence on the calibration.

Erroneously high neutron counts may be caused by water or other hydrogen-bearing material near the measure and probe. It was noted that the readings were increased if the operator stood near the measure. Similarly, water on the floor or in the earth beneath the measure can be detected and will increase neutron count readings. The influence of temperature on the apparatus is another source of discrepancy; calibrations should be checked for the temperature at which the apparatus will be used. It is possible under some conditions of local electrical static that the electrical shielding of the amplifier may be insufficient, and false counts may be produced.

The results reported here are encouraging, but somewhat better precision than was found in these tests is desirable. Improved procedures and apparatus are needed to provide better protection from external influences, calibration that accounts for the effects of elements other than hydrogen, and arrangement of the sample, source, and detector to eliminate local effects caused by the position of aggregate particles.

ACKNOWLEDGMENT

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CORROSION AND SERVICE LIFE OF
CORRUGATED METAL PIPE IN KANSAS

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Nearly 2,000 corrugated metal pipes as old as 43 years were inspected in a research study in Kansas during a period of 6 years. Bituminous coatings applied to pipes seldom remained intact in exposed areas for more than 3 or 4 years; sometimes they were intact for only a few months. Consequently, they are not of sufficient value to justify their use in Kansas. Plain galvanized corrugated metal pipes without bituminous coatings have served well in Kansas for more than 40 years. A few pipes in localized strip pit coal mining areas have corroded excessively, but none has failed structurally. Aluminum pipes are in good condition but had been used in Kansas for only 7 years at the time of this study, and none was installed near coal mines.

●CORRUGATED metal pipes have been used successfully in Kansas for more than 40 years. Installations were made at farm and field entrances, for erosion control, and more recently on side roads and across the centerline of main highways. This report summarizes research studies of corrugated metal pipe recently conducted by the State Highway Commission of Kansas (1, 2, 3).

Figure 1 shows the 35 counties in which plain galvanized steel pipes were inspected in 1965, 1967, and 1968 to determine corrosion and service life. An investigation was conducted in 1969 to determine the effectiveness of bituminous coatings on corrugated metal pipes, both aluminum and galvanized steel. These locations included 53 counties that were well distributed throughout the state. Aluminum pipes have not been used long on the state highway system. They were used in 34 counties during the period from 1963 to 1966 (designated A, Fig. 1). Inspection of these was delayed until 1970 to provide more time for evaluation of performance.

PROCEDURE

Office records were searched to find the locations where corrugated metal pipes were used. The round pipes varied in diameter from 15 to 54 in.; 92 percent of them were 24 in. or less. The smaller pipes were usually 16 gauge, and the larger ones were either 14 or 12 gauge. The base metal was usually copper steel, galvanized on both sides by the hot-dip process with no less than 2 oz/ft^2 of double exposed surface. Pipes were inspected and certified at the time of construction but not verified during our investigation. From 2 to 6 pipes on each project were inspected in the field and rated according to the following system:

Condition	Rating
No corrosion, galvanizing intact	0
Superficial rust (edge and bolt heads), no pitting, weathered, zinc luster gone	1
Moderate rust, rust flakes tight, minor pitting	2
Fairly heavy rusting, rust flakes tight, moderate pitting, metal sound	3

Condition	Rating
Heavy rusting, rust flakes easily removed, deep pitting into base metal	4
Heavy rust, deep pitting, perforated areas, unsound areas easily perforated with pick end of geologist hammer	5

The Hoffer test (4) was run on each soil sample obtained in 1967 to determine whether oxidizing or reducing conditions existed. Inasmuch as no significance was found, the test was not continued. The problem of abrasion was checked in the field, but only one pipe was found to be carrying much rock debris. That pipe was not abraded. A sample of soil from each steel pipe location was taken to the laboratory for determination of pH and soil resistivity. If water was standing in or flowing through a pipe, the water was sampled and taken to the laboratory for pH measurements. The water samples were always tested immediately after they were received in the laboratory in order to prevent possible changes in pH values resulting from biological growth or other changes that might occur in the water.

The percentage of aluminum pipes inspected was based on the number of pipes in each soil group. All of the pipe locations were numbered, and a table of random numbers was used to determine the 260 pipes to be inspected from the total of 921 that were installed from 1963 to 1966. Aluminum pipes are still permitted, but contractors have not elected to use them on the state highway system since that date. Aluminum pipes inspected were aluminum alloy conforming to AASHO Designation M 196.

Specifications called for the bituminous coatings to be at least 0.03 in. thick and to consist of asphalt cement 99.5 percent soluble in carbon bisulfide. Certification was required for stability, imperviousness, and resistance to impact and erosion.

Preliminary investigation of the bituminous-coated pipes revealed that bare galvanizing is usually exposed in fewer than 4 years after installation. Records were searched to find locations where bituminous-coated corrugated metal pipes were installed in 1965 and 1966. In addition, a few coated pipes were inspected that had been in place from 10 to 32 years to determine whether coating had any significant effect on the galvanizing or corrosion and whether perchance some of the older coatings may yet be good.

Inspections were made primarily of the inside of the pipes, where previous investigations in Kansas and elsewhere have shown that corrosion is most destructive. Graf, James, and Wendling (3) found in their study of pipe culvert corrosion that in almost all cases the invert of the pipe had the highest degree of corrosion. Studies in Kansas showed that abrasion was no problem and, therefore, would not cause invert deterioration. Haviland et al. (5) found that metal loss associated with corrosion originates on the culvert's interior surface and progresses toward the exterior surface. This was true for the inverts as well as for other portions of the pipe. Holt (6) and Malcom (7) state that strip corrosion, which can ultimately cause structural inadequacy, occurs from chemical corrosion on the interior of culverts from exposure to corrosive waters and not from galvanic spot corrosion on the exterior. Random perforations from localized spots were not found to connect to cause strip corrosion. In Nebraska, Bearg et al. (8) cut plug samples out of several pipes and found that in no case was there any pitting or measurable metal loss from the outside even though some of the culverts appeared to be deteriorated from the inside. Such research findings showing that most corrosion begins on the inside of the pipe indicated that there was no reason to remove the earth fill from the pipes to determine the condition of the bituminous coatings on the outside.

RESULTS ON BITUMINOUS-COATED PIPES

Table 1 gives a summary of results for pipes 3 and 4 years of age. The coatings were considered good only where no bare galvanizing was observed. This condition was found only on 12 percent of the pipes inspected. Most of the good coatings were found in storm sewer installations where the weather conditions are less severe. Even with these better conditions, bare galvanizing was usually exposed, leaving only 27

percent of the storm sewers with good coatings. When the storm sewer pipes are sub-
tracted from the total number of pipes inspected, only 6 percent of the others have good
coatings. Ten bituminous-coated aluminum pipes were inspected. The coatings on
these pipes were in the same condition as that of the steel pipes.

The preceding discussion applies only to pipes 3 and 4 years old. Bare galvanizing
was exposed at least near the outlet on all of the older pipes inspected, including storm
sewers. Corrosion was not significantly different from that observed on uncoated steel
pipes.

Figure 2 shows a pipe with strip bare in the invert. Figure 3 shows a pipe with about
half of the coating gone. Sometimes the coatings were only cracked, and in a few cases
they were entirely good.

Some bituminous coatings disappear during construction. Before grass is seeded
and develops a sod cover, mud from bare back slopes, ditch slopes, and ditch bottoms
is carried into newly installed pipes. Cohesive soil adheres to the bituminous coatings,
and, when the soil dries and shrinks, it pulls the bituminous coating from the pipe. This
has occurred without freezing and thawing, both in the field and in laboratory tests.
Freezing and thawing has also removed bituminous coatings. Damage has occurred
under both of these conditions at the end of the second cycle.

Some other states have indicated that bituminous coatings are helpful. Haviland et
al. (5) reported that coated culverts included in a New York investigation had only 62
percent as much metal loss as uncoated culverts but that more than 40 years of ser-
vice can be expected for most of their uncoated culverts. Beaton and Stratfull (9) in
California reported that the actual added life varied from nearly 0 years in areas of
continuous flow carrying heavy debris to more than 20 years in arid areas with infre-
quent runoff; an average of 6 years was added by bituminous coatings. In North Caro-
lina, Welborn and Serafin (10) noted that the better condition of the asphalt coating on
one section as compared with another seems to indicate that the coating's durability
depends considerably on the type of asphalt used. The asphalt coating on the outside
of the pipe usually exhibited a checked pattern of cracking and was found to have very
little adhesion to the metal. Coated pipe that lost the coating early in service showed
approximately the same amount of corrosion as uncoated pipe. In Kansas we have found
that our bituminous coatings are of little benefit.

RESULTS ON ALUMINUM PIPES

The aluminum pipes were all good and had no pitting. Many of the pipes were clean,
with no accumulation of soil. Soil samples were obtained from 23 of the 260 pipes in-
spected. The values of electrical resistivity and pH are given in Table 2. All pH values
are in the range favorable to aluminum as well as to steel.

RESULTS ON STEEL PIPES

The number of steel pipes found with each rating is as follows:

Rating	Number		Rating	Number
0	11		3	176
1	455		4	55
2	213		5	19
			Total	929

Only a relatively few pipes had more than moderate rust with minor pitting. Table
3 gives the data collected in one county; there are 59 pages more of such tables.

A chart was developed in California (11) several years ago for estimating years to
perforation based on pH and electrical resistivity of the surrounding soil. It was an-
ticipated that the chart could be adjusted for conditions in Kansas. Our investigation
of pipes from 7 to 43 years old revealed no correlation of pipe condition with pH and
electrical resistivity. We had a relatively narrow range of values of pH, usually be-
tween 5.4 and 8.5, and little significance was attached to individual values for differences

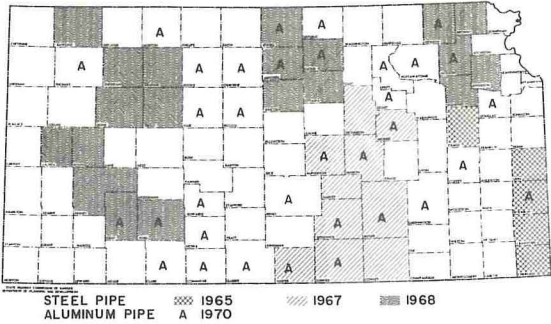## Figure 1. Counties in Kansas where pipes were inspected.

STEEL PIPE   ▓ 1965   ╱ 1967   ▓ 1968
ALUMINUM PIPE   A 1970

Table 1. Bituminous-coated pipes 3 and 4 years old.

Division	Total Pipes Inspected	Coatings Good	Storm Sewer Pipes Inspected	Coatings Good
1	133	9	42	8
2	57	4		
3	105	16	15	8
4	74	1	27	1
5	133	13	42	8
6	66	24	24	16
Total	568	67	150	41

Figure 2. Bituminous coating in 8-in. wide strip on 2-ft pipe gone in 3 years.

Figure 3. Bituminous coating on half of 2-ft pipe gone in 3 years.
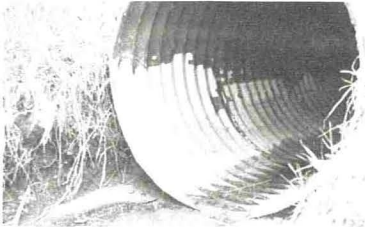
Table 2. Soil samples for aluminum pipes.

County	Station	Side	Use	Size (ft)	Resistivity	pH
Barber	62+30	Left	Entrance	2 by 24	4,315	8.3
Coffey	177+75	Right	Entrance	1.5 by 26	732	7.8
	153+93	Left	Entrance	1.5 by 26	1,050	7.9
	118+40	Right	Entrance	1.5 by 26	719	7.3
Commanche	291+80	Left	Entrance	7 by 40	1,145	8.3
Douglas	566+33	Left	Entrance	1.5 by 26	910	8.2
Edwards	7+00	Centerline	Crossroad	2 by 48	6,129	7.0
Geary	823+50	Right	Ramp	6 by 32	1,204	8.0
Gray	840+00	Centerline	Crossroad	6.8 by 40	1,976	8.1
	104+65	Right	Entrance	2 by 30	1,251	8.0
Greenwood	157+00	Right	Entrance	2.8 by 24	878	7.8
	95+51	Right	Entrance	1.5 by 24	876	7.8
Norton	42+35	Right	Entrance	2 by 36	1,301	8.6
Osborne	118+60	Right	Entrance	2 by 36	956	8.0
	156+80	Right	Entrance	2 by 40	813	8.0
Pottawatomie	202+10	Right	Entrance	2 by 20	1,247	8.0
	8+58	Left	Entrance	1.5 by 30	863	7.9
	572+95	Left	Entrance	2 by 24	976	8.5
Reno	1024+90	Right	Entrance	2 by 24	1,503	8.0
Riley	77+00	Left	Entrance	2 by 24	1,119	8.2
Russell	308+86	Left	Entrance	2 by 44	2,174	8.0
	226+92	Left	Entrance	2 by 32	1,008	Lost
	952+34	Right	Entrance	2 by 32	1,224	8.0

Table 3. Summary of pipe condition survey in Morris County.

Station	Diameter (ft)	Sample	Soil From	Soil pH	Resistivity	Hoffer Color[a]	Water pH[b]	Age 1967	Predicted Life Actual Pipe	16-Gauge Pipe	Present Rating
51+00	4.5	101	Fill	7.4	1,865	Red		20	53	41	1
51+00[c]	4.5	102	Invert	7.7	1,215	Red		20	46	35	1
58+35	2.5	103	Fill	7.9	1,535	Red		20	49	38	2
58+35[c]	2.5	104	Invert	8.0	1,236	Red		20	47	36	2
61+70	2.5	105	Invert	7.8	1,055	Red		20	44	34	2
180+78	43.0	106	Fill	7.9	1,236	Red	7.8	20	83	36	1
408+50	1.5	107	Fill	7.7	1,008	Red		37	34	34	2
408+50[c]	1.5	108	Invert	7.5	1,055	Red		37	34	34	2
483+30	1.5	109	Invert	7.5	1,301	Red		37	36	36	3
483+30[c]	1.5	110	Fill	7.5	1,567	Red		37	38	38	3
501+37	1.5	111	Invert	7.5	1,724	Red		37	40	40	2

[a]Red indicates oxidizing conditions; blue indicates reducing conditions.
[b]Absence of values usually indicates intermittent flow and no water at the time of sampling.
[c]Same pipe as the one listed at this location in preceding line.

within this range. Neither was significance found for variation in resistivities that were generally between 700 and 2,300.

Evaluations were made for conditions that existed at the time of sampling. There is no doubt, for the older pipes at least, that environmental conditions have changed during the life of the pipes. For instance, the possible effect of chemical fertilizers and atmospheric and surface pollution of recent years probably is not much reflected in the present pipe life. It may be that galvanized pipe reaches a state of equilibrium with the soil and thereafter changes very slowly in the process of perforation. Hard water tends to produce a protective coating, and sometimes a pipe will have a buildup of iron rust from the soil and will not pit and perforate.

Several counties other than those shown in Figure 1 have had coal produced in them, and a study was made to ascertain the conditions in some of these areas. Usually the veins of coal are 14 in. or less in thickness and were mined from 40 to 60 years ago. No evidence was found that any of these old abandoned mine sources had affected pipes. However, another research study made near a current coal production site revealed that the entire invert of a normal galvanized steel test pipe was destroyed by corrosion in less than 1 year. The California chart also indicated a life of less than 5 years in this instance. In this same area, an entrance pipe had previously been replaced as a result of corrosion, the only case known in the entire state. Other pipes had been replaced for other reasons, and some of them have been slightly or severely corroded; but, as far as we know, no other pipe has been replaced on the state system of highways primarily because of corrosion. These studies show the importance of checking the geology reports and observing in the field the presence or absence of coal mines or coal seams. In case of doubt, especially if replacement would be expensive or difficult, perhaps metal pipes should not be used. Otherwise, long service life may be expected.

SUMMARY AND CONCLUSIONS

Variations of either pH or resistivity had little effect on corrugated metal pipe life in Kansas. All but 4 measured values of pH were between 5.4 and 8.5, and 90 percent of resistivities were between 700 and 2,300. Within these ranges, corrosion was not sufficient to determine a correlation such as that indicated by the California chart. In 3 surveys of galvanized steel pipes covering 43 years of service in 35 counties well distributed in the state, 929 pipes were inspected and only 19 perforated ones were found, most of which were in the vicinity of coal mines. All of them were serviceable. None had failed structurally.

This record of service indicates that corrugated metal pipes with normal galvanizing are satisfactory for most of Kansas except in the immediate vicinity of coal mines. No soil series was found that indicated that special pipes are needed. There are no entire counties or large areas of a county in Kansas where metal pipes need to be precluded. Additional attempts to designate critical areas are not practical. The presence or absence of coal mines in the vicinity of any project can be observed. A life of 40 to 50 years or more may be anticipated for normal galvanized steel pipe in Kansas at most locations other than near active coal mines.

Bituminous coatings on the inside of corrugated metal pipes in Kansas are usually cracked and partially lost in less than 3 years. In many cases the coatings are lost in only a few months. The condition of the bituminous coating on the buried portion of the outside of the pipe is not significant, because destructive strip corrosion originates on the interior surface and progresses toward the exterior surface. No noticeable corrosion was found on any of the aluminum pipes, all of which were 7 years or less in age.

IMPLEMENTATION OF RESULTS

The average annual expenditure for bituminous coating of metal culvert pipes in Kansas (computed as the additional cost for coated pipe over an equivalent amount of plain galvanized metal pipe and including costs for inspection of the coating) is approximately $25,000. This investment generally buys less than 3 years of coating life and probably no added pipe life. It is recommended that bituminous coatings be discontinued on metal pipes for highway construction in Kansas.

This investigation has shown that galvanized steel pipes may be used on any highways in Kansas and that no serious problems exist except as noted in connection with the mining of coal, which in Kansas is usually a strip pit operation. Otherwise galvanized steel pipes may be expected to last 40 to 50 years or longer. The geology report may be checked for the possibility of coal on or near a project. The presence of any detrimental coal mining waste or debris may be observed in the field during the design process, or it may be requested to be included in the geology or soils reports.

Inasmuch as the deleterious effluent from the coal mining areas contains sulfuric acid, we recommend that any pipe used in these areas where conditions are the worst be constructed of sulfate-resistant materials as recommended by Krizek et al. (12). Additional protection may be achieved by lining the ditch with broken limestone and using limestone screenings for backfill around the pipe (13, 14). The pollution abatement program of the state may eventually reduce or eliminate the sources of pollution from active coal mines (15).

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COMPARATIVE INVESTIGATIONS OF INTERNATIONAL TEST METHODS FOR SMALL-SIZED COARSE AGGREGATES

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Not enough detailed knowledge exists about loads and stresses in road pavements. Consequently, based on certain models, there are many different test procedures for coarse aggregates that strain the materials by compression, impact, and abrasion or by a combination of these stresses. A comparison of test values that are obtained with the different test methods has not been possible, and experiences with various qualified aggregates cannot be transmitted from one country to another. Comparative tests were conducted by using different aggregates with the most important test methods: Los Angeles, Deval, German impact, British impact, and modified Marshall impact in standardized and modified form. The calculated linear regressions showed that, although the strains of the aggregates are very different, the tests characterize the same or at least similar properties of aggregates. In a comparison of different laboratories, we obtained a relative reproducibility of about 20 to 30 percent for the Los Angeles and the German impact tests. This means that further strict standardization of testing machinery and procedure is necessary if values obtained at different laboratories are to be compared. The calculated linear regressions that were found for the different test procedures will allow the comparison of specifications for small-sized coarse aggregates tested with different international test methods. International experiences and literature on the question of requirements for use of aggregates can now be exploited.

•NOT enough detailed knowledge exists on the loads and stresses in road surfaces resulting from traffic. Consequently, no exact values are known about the nature and the quantity of stress that highway materials are undergoing. Based on certain models, many different test procedures have been developed for coarse aggregates used in road surfaces. The specifications for their use in different countries are largely based on subjective experiences and differ greatly. The test procedures developed for testing coarse aggregates stress the materials by compression, impact, and abrasion or by a combination of these stresses. In no case, however, can a detailed analysis of stresses be performed, and thus an exact and objective evaluation of test results is not possible. A comparison of test values obtained with the different test methods has also not been possible. Because test methods are different, the experiences with various satisfactory aggregates cannot be transferred from one country to another. A meaningful comparison of national requirements for special use of aggregates has also been impossible.

In West Germany the standardized impact test is strongly questioned, especially because of the great variation in test results obtained when identical materials are tested in different laboratories. Comparative tests have shown relative deviations of ±20 percent. We believed we could find better methods that we could standardize in the future.

INTERNATIONAL TEST METHODS FOR COARSE AGGREGATES

A detailed study of the international literature on test methods for small-sized coarse aggregates shows that special test procedures are used that stress the material by compression, impact, and abrasion or by a combination of these stresses. A procedure with compressive load for small-sized coarse aggregate is only standardized in the British Standard 812 (28) and for large-sized coarse aggregate in the German DIN 52 109 (29).

Test procedures with abrasive strain include the Deval test (22, 25, 26, 37), which is of French origin (3, 4). The most popular abrasive method is the American Los Angeles test that is used in its original or modified form in many countries around the world, for example, in the United States (23, 24), France (36), Sweden (11), Hungary (16, 20), and the Soviet Union (5). Impact crash is used for testing coarse aggregate in Norway (8), Sweden (11), Great Britain (28), Switzerland (10, 21), and Germany (29, 30, 33). The German impact test had its origin in testing methods for large-sized coarse aggregate (29) and was later modified for small-sized coarse aggregate (30, 33) in a size of 8 to 12.5 mm ($\approx\frac{5}{16}$ to $\frac{1}{2}$ in.). This test method has long been questioned. The test machine for impact crash is very complex, and as a result only a small number of testing laboratories are equipped with it. For many years, we have tried to use equipment that is simpler and more often available such as the Marshall compaction hammer (27). The research of Pös (17) and Gragger (7) has shown that such a modified test method will bring sufficient correlation with the usual German impact test so that its use for quality control in quarries or at hot-mix plants is possible.

For testing small- and large-sized coarse aggregates, we believe that 5 testing methods are of special interest from an international point of view: the American Los Angeles test, the French Deval test, the British impact test, the German impact test, and the modified Marshall impact test.

Only a few mathematical relations among these 5 procedures can be found in the literature. Höbeda (11) reported on relations among the Swedish impact test, the German impact test, and a modified Los Angeles test. Nagel (14) found linear regressions among the German impact test, a modified Los Angeles test, and a compression test for coarse aggregate of size $\frac{5}{16}$ to $\frac{1}{2}$ in. and found linear correlation coefficients in the range of 0.89 to 0.96. Brand and Dinkgraeve (1) conducted comparative researches with the Los Angeles test (23, 24) and the German impact test (30, 33) for large- and small-sized coarse aggregates and found linear correlation coefficients of 0.90 and more. In Gragger's work (7), a correlation coefficient of 0.98 between results of the German impact test and the modified Marshall impact test was found.

The relations given above show that even test methods with very different loads and procedures will identify similar properties of aggregates. It was the main task of this research program to analyze experimentally such relations among the most important test methods, to point out the value of the test results, and to calculate the variations among the different methods.

TEST PROCEDURES

Initially using the 5 basic test methods in 23 standardized and modified procedures, we made 10 tests of each with 2 aggregates—limestone and basalt. The tests were only performed with methods that are used for small-sized coarse aggregates, 1 in. to No. 8, inasmuch as larger sizes usually are not used in the surface and base course in road construction. After calculating the dispersions (standard deviations), we eliminated 12 modified procedures that showed large standard deviations under identical conditions.

A full test program was then conducted with the remaining 11 test procedures. The test conditions are given in Table 1.

Test method 1 represents the German impact test (30, 33) with aggregate of size 8 to 12.5 mm ($\approx\frac{5}{16}$ to $\frac{1}{2}$ in.) in a modified form. We took the percentage passing the 3.15-mm ($\approx\frac{1}{8}$ in.) sieve for calculating the impact value, SD_{Sp8} (14, 15), instead of using the 5 sieves (8, 5, 2, 0.63, and 0.2 mm) for analyzing an impact crash value, SZ_{Sp} (33). Nagel (15) found a high correlation coefficient for a linear regression between SD_{Sp8} and SZ_{Sp} as shown in Figure 1. The test apparatus is shown in Figure 2. The

weight of the sample was $0.5 \times \rho_R$, where ρ_R = bulk specific gravity of aggregate in g/cm^3. The material was planed in a cylindrical steel cup and received 10 blows with a 110-lb hammer dropping from a 19.7-in. height.

For calculating the abrasion with the Los Angeles machine, the gradings B, C, and D of test samples as given in ASTM Designation C 131-69 (23) were used. In addition to these standardized methods, the gradings were varied corresponding to the German test materials of size 8 to 12.5 mm ($\approx$ $^5/_{16}$ to $^1/_2$ in.). The test value LD^*_{Sp8} is obtained by using a weight of 5,000 g and analyzing the abrasion value as the percentage passing the 3.15-mm ($\approx$ $^1/_8$ in.) sieve. The test value LD_{Sp8} is based on a sample with a weight of 2,500 g.

From the Deval test, the gradings D and E of ASTM Designation D 289-63 (26) were selected. Aside from these standard methods, no modified procedures were tested. We had learned from our first test program that with the Deval test procedure sufficient spreading cannot be achieved to differentiate "abrasive" from "nonabrasive" aggregates.

The modification of the British impact test had shown high dispersions in the first program so that only the standard material ($^1/_2$ to $^3/_8$ in.), according to British Standard 812 (28), was tested. The quantity of test material is determined by a "measure shell" and, depending on the bulk specific gravity of aggregate, amounts to 340 to 400 g. The test sample is subjected to 15 blows by a hammer of 30 to 31 lb falling freely from 15 in. (Fig. 3). The test value ID_{Bs} is calculated as the percentage passing sieve No. 7 (BS).

For the modified Marshall impact test, the Marshall compaction hammer (27) is used. The test value MD^*_{Sp8} is obtained when coarse aggregate of size $^5/_{16}$ to $^1/_2$ in. with a weight of $260 \times \rho_R$ (in g) is compacted by 200 blows. How the material is put into the 4-in. mold cylinders between 2 metal plates is shown in Figure 4. A second Marshall value, MD_{Sp8}, is obtained by the use of a sample weight of 400 g. Both values are calculated as the percentage passing a 3.15-mm ($\approx$ $^1/_8$ in.) sieve.

The method of sampling, preparing, and grading guaranteed that all samples were members of one population, thus permitting a statistical evaluation.

For all test procedures, materials of a shape with a breadth-thickness ratio of b:d $\leq$ 2 were prepared by a second sieve analysis with so-called slit sieves (14, 18) after narrow ranges were graded with usual sieves.

With each of these 11 procedures, 15 different aggregates that are used in road construction were tested. The program included 11 igneous rocks (granites, diabases, and basalt), a metamorphic rock (quartzite), and 3 sedimentary rocks (2 different limestones and a quartzitic sandstone).

ANALYSIS OF REGRESSION

With each of the 11 test methods and all 15 aggregates, 3 tests were made. The mean value of the 3 single values was the input for the analysis of regression (9). By the use of linear regression $y = a + bx$, the correlation coefficient for the relation between each two test procedures could be calculated (6, 9, 21).

The correlation coefficients r_{xy} for the linear regressions between the different test methods are given in Table 2. The calculated equations for the linear regressions are given in Table 3. The equation given in Table 3 that shows the linear regression for any correlation of 2 test methods is also given in Table 2.

A test developed by Fisher (6) can be conducted to test whether the correlations are significantly greater than 0. With the value

$$t = \left(r_{xy} \Big/ \sqrt{1 - r_{xy}^2} \right) \cdot \sqrt{n - 2}$$

or

$$r_{xy} = t \Big/ \sqrt{t^2 + n - 2}$$

the confidence belts for r_{xy} can be calculated for given reliabilities. The t-statistic is tabulated for several reliabilities and the number of degrees of freedom f. With the

Table 1. Test conditions.

Test Method	Value	No.	Size Fractions	Weight of Indicated Sizes	Hammer Weight (lb)	Blows	Free Fall (in.)	Revolutions	Spheres Number	Spheres Weight (g)	Test Sieves
German impact	SD_{SpB}	1	$5/16$ to $1/2$ in.	$0.5\rho_R^a$	110	10	19.7	—	—	—	DIN 3.15 mm
Los Angeles	$LD_{B/ASTM}$	2	$3/4$ to $3/8$ in.	5,000 g	—	—	—	500	11	4,584	ASTM No. 12
	$LD_{C/ASTM}$	3	$3/8$ to No. 4	5,000 g	—	—	—	500	8	3,330	ASTM No. 12
	$LD_{D/ASTM}$	4	No. 4 to No. 8	5,000 g	—	—	—	500	6	2,500	ASTM No. 12
	LD_{SpB}^*	5	$5/16$ to $1/2$ in.	5,000 g	—	—	—	500	12	5,000	DIN 3.15 mm
	LD_{SpB}	6	$5/16$ to $1/2$ in.	2,000 g	—	—	—	500	6	2,500	DIN 3.15 mm
Deval	$VD_{D/ASTM}$	7	$3/4$ to No. 4	5,000 g (5,500 g)	—	—	—	10,000	6	2,500	ASTM No. 12
	$VD_{E/ASTM}$	8	$1/2$ to No. 4	5,000 g (5,500 g)	—	—	—	10,000	6	2,500	ASTM No. 12
British impact	ID_{BS}	9	$1/2$ to $3/8$ in.	340 to 400 g	30 to 31	15	15	—	—	—	BS No. 7
Modified Marshall impact	MD_{SpB}^*	10	$5/16$ to $1/2$ in.	$0.26\rho_R^a$	10	200	18	—	—	—	DIN 3.15 mm
	MD_{SpB}	11	$5/16$ to $1/2$ in.	400 g	10	200	18	—	—	—	DIN 3.15 mm

^{a}Bulk specific gravity.

Figure 1. Correlation of German impact values SD_{Sp8} and SZ_{Sp}.

$SD_{Sp8} \begin{bmatrix} percent \\ weight \end{bmatrix}$

$SD_{Sp8} = -3.99 + 1.14 \cdot SZ_{Sp}$
$r_{xy} = 0.99$

$SZ_{Sp} \begin{bmatrix} percent \\ weight \end{bmatrix}$
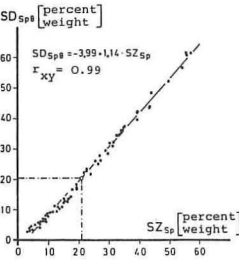
Figure 2. Apparatus for German impact test.

tup guide bars

hammer (tup) free fall 19.7 in (weight 110 lb)

case hardened surface

cylindrical steel cup

steel base (weight 1100-1320 lb)
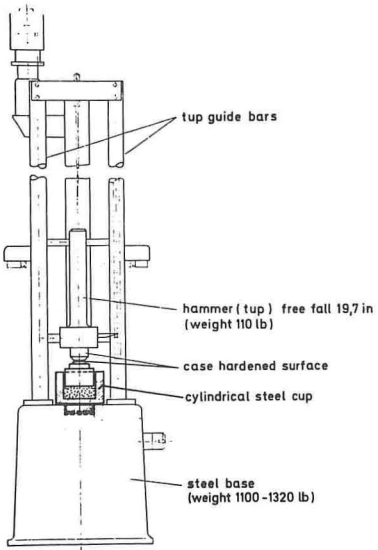
Figure 3. Apparatus for British impact test.

Locking pin for release mechanism

Adjustable stop for release

Lifting handles

Ratchet counter, (to count number of blows)

Release claw

Tup (weight 30-31 lb)

$1/16$ in chamfer

Case-hardened surface

$3^{15}/16$ in dia

$15 \pm 1/4$ in

Tup guide bars

4 in dia

Cylindrical steel cup, inner surfaces case hardened

Circular base
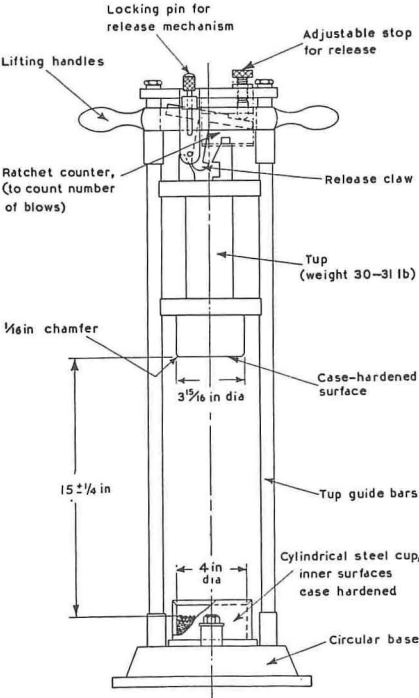
sample size n = 15 and f = n - 2 = 13, the confidence belts for the significance of the correlation are as follows:

Reliability (percent)	Confidence Belt for r_{xy}
95	0.51
98	0.59
99	0.64
99.9	0.75

Data given in Table 2 show that only 2 of the 55 values of the correlation coefficient r_{xy} are less than 0.75 and both are greater than 0.64. That means that in all tested cases the calculated linear regression exists with a reliability of more than 99 percent and mostly with more than 99.9 percent. It is concluded that all correlations between testing methods for coarse aggregates are significant. This would mean that with the different test methods and values the same or at least similar properties of aggregates are characterized.

With correlation coefficients close to 1, the correlations have increasing significance. Thus, we can see several special results (Table 2). Relations between procedures with similar loads show the highest correlation coefficients. This can be seen in a comparison of the German impact test, the British impact test, and the modified Marshall impact test with one another and the Los Angeles test and the Deval test with each other. As shown by the confidence belts, however, the correlations comparing the impact methods with the abrasion methods are also highly significant. Representative of that is the regression of test 1, the German impact test SD_{sp8}, and test 2, the Los Angeles test grading B, as shown in Figure 5.

An interesting phenomenon that we observed is that the correlation coefficients decrease as differences of grading size of tested aggregates increase. This is very significant with the relation of the German impact test to B, C, and D gradings of the Los Angeles Test.

Grading B ($^3/_4$ to $^3/_8$ in.) has a size similar to the grading of the German impact test ($^5/_6$ to $^1/_2$ in.) and thus $r_{xy} = 0.95$. The relations of the C and D (with different sized fractions of $^3/_8$ to No. 4 and No. 4 to No. 8) to the German impact test are $r_{xy} = 0.87$ and 0.75 respectively.

This phenomenon was also reported by Nagel (14), who showed that the behavior of smaller sized fractions with the German impact test depends on the different types of aggregate, as Figure 6 shows.

If the value for fraction size 8 to 12.5 mm ($^5/_{16}$ to $^1/_2$ in.) is set equal to 1.0, the relative value SD_{sp1}/SD_{sp8} differs greatly, particularly for the smaller gradings (size i in Fig. 6). Some aggregates (C and D in Fig. 6) show relatively smaller values (<1.0); others (A and B) show relatively larger values (>1.0). That means that the smaller sizes of several aggregates have an increasing tendency to be crushed by impact. Nagel (14) called this a "selection," which means that in some aggregates one finds relatively weak particles in the smaller sizes. So it is sometimes difficult to estimate the behavior of an aggregate in all sizes if values for only one size are obtained. This different behavior of aggregates may be an explanation for the decreasing significance of correlation in relating test procedures with different sizes of test materials.

ANALYSIS OF VARIANCE

With the single values of random samples, we could calculate the variance (9) of each test procedure. With the different tested aggregates, we had k = 15 sets with n = 3 single values to conduct an analysis of variance for calculating variation within sets (9).

The variations are given in Table 4. The standard deviations calculated from n = 10 single values for a basalt and a limestone are listed for comparison. With the Los Angeles and Deval tests (No. 2 through No. 8), we generally found smaller deviations than with the German test (No. 1). In most cases we found significantly larger deviations with the British and the modified Marshall tests (No. 9, No. 10, and No. 11).

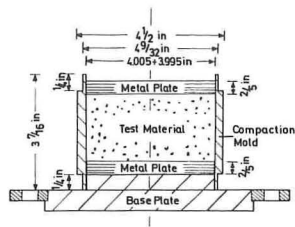**Figure 4. Mold cylinders with aggregate for modified Marshall test.**

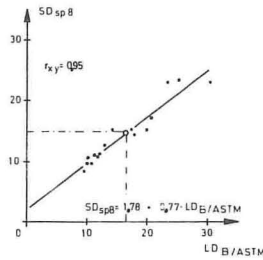**Figure 5. Linear regression $SD_{Sp8}/LD_{B/ASTM}$.**

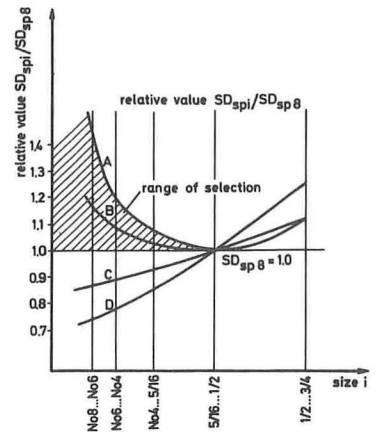**Figure 6. Relative impact value versus size of coarse aggregate.**

Table 2. Correlation coefficients for linear regression.

Test	Test 1	2	3	4	5	6	7	8	9	10	11
1		1:2	1:3	1:4	1:5	1:6	1:7	1:8	1:9	1:10	1:11
2	0.95		2:3	2:4	2:5	2:6	2:7	2:8	2:9	2:10	2:11
3	0.87	0.96		3:4	3:5	3:6	3:7	3:8	3:9	3:10	3:11
4	0.75	0.87	0.96		4:5	4:6	4:7	4:8	4:9	4:10	4:11
5	0.93	0.99	0.98	0.91		5:6	5:7	5:8	5:9	5:10	5:11
6	0.95	0.99	0.96	0.88	0.99		6:7	6:8	6:9	6:10	6:11
7	0.83	0.93	0.98	0.96	0.95	0.92		7:8	7:9	7:10	7:11
8	0.81	0.92	0.98	0.94	0.94	0.90	0.98		8:9	8:10	8:11
9	0.96	0.96	0.92	0.80	0.95	0.95	0.89	0.89		9:10	9:11
10	0.94	0.92	0.82	0.68	0.88	0.91	0.76	0.75	0.92		10:11
11	0.94	0.95	0.86	0.74	0.92	0.94	0.79	0.80	0.92	0.97	

Table 3. Equations for linear regressions and correlation coefficients.

Regression	Equation	r_{xy}	Regression	Equation	r_{xy}
1:2	$SD_{Sp8} = 1.78 + 0.77\ LD_{B/ASTM}$	0.95	4:6	$LD_{D/ASTM} = 7.22 + 1.00\ LD_{Sp8}$	0.88
1:3	$SD_{Sp8} = 2.00 + 0.65\ LD_{C/ASTM}$	0.87	4:7	$LD_{D/ASTM} = 7.79 + 1.76\ VD_{D/ASTM}$	0.96
1:4	$SD_{Sp8} = 2.14 + 0.52\ LD_{D/ASTM}$	0.75	4:8	$LD_{D/ASTM} = 8.45 + 1.97\ VD_{E/ASTM}$	0.94
1:5	$SD_{Sp8} = 2.43 + 0.45\ LD_{Sp8}^{*}$	0.93	4:9	$LD_{D/ASTM} = 13.18 + 1.04\ ID_{Bs}$	0.80
1:6	$SD_{Sp8} = 2.00 + 0.75\ LD_{Sp8}$	0.95	4:10	$LD_{D/ASTM} = 16.60 + 0.91\ MD_{Sp8}^{*}$	0.68
1:7	$SD_{Sp8} = 4.83 + 1.08\ VD_{D/ASTM}$	0.83	4:11	$LD_{D/ASTM} = 14.96 + 0.62\ MD_{Sp8}$	0.74
1:8	$SD_{Sp8} = 5.26 + 1.19\ VD_{E/ASTM}$	0.81	5:6	$LD_{Sp8}^{*} = 0.06 + 1.60\ LD_{Sp8}$	0.99
1:9	$SD_{Sp8} = 5.57 + 0.87\ ID_{Bs}$	0.96	5:7	$LD_{Sp8}^{*} = 3.91 + 2.49\ VD_{D/ASTM}$	0.95
1:10	$SD_{Sp8} = 7.48 + 0.87\ MD_{Sp8}^{*}$	0.94	5:8	$LD_{Sp8}^{*} = 4.52 + 2.83\ VD_{E/ASTM}$	0.94
1:11	$SD_{Sp8} = 6.66 + 0.55\ MD_{Sp8}$	0.94	5:9	$LD_{Sp8}^{*} = 8.05 + 1.75\ ID_{Bs}$	0.95
2:3	$LD_{B/ASTM} = -0.59 + 0.88\ LD_{C/ASTM}$	0.96	5:10	$LD_{Sp8}^{*} = 13.16 + 1.67\ MD_{Sp8}^{*}$	0.88
2:4	$LD_{B/ASTM} = -1.44 + 0.75\ LD_{D/ASTM}$	0.87	5:11	$LD_{Sp8}^{*} = 10.87 + 1.09\ MD_{Sp8}$	0.92
2:5	$LD_{B/ASTM} = 0.51 + 0.59\ LD_{Sp8}^{*}$	0.99	6:7	$LD_{Sp8} = 2.98 + 1.49\ VD_{D/ASTM}$	0.92
2:6	$LD_{B/ASTM} = 0.30 + 0.97\ LD_{Sp8}$	0.99	6:8	$LD_{Sp8} = 3.53 + 1.68\ VD_{E/ASTM}$	0.90
2:7	$LD_{B/ASTM} = 3.10 + 1.46\ VD_{D/ASTM}$	0.93	6:9	$LD_{Sp8} = 5.49 + 1.08\ ID_{Bs}$	0.95
2:8	$LD_{B/ASTM} = 3.45 + 1.67\ VD_{E/ASTM}$	0.92	6:10	$LD_{Sp8} = 7.97 + 1.07\ MD_{Sp8}^{*}$	0.91
2:9	$LD_{B/ASTM} = 5.43 + 1.07\ ID_{Bs}$	0.96	6:11	$LD_{Sp8} = 6.62 + 0.70\ MD_{Sp8}$	0.94
2:10	$LD_{B/ASTM} = 7.96 + 1.05\ MD_{Sp8}^{*}$	0.92	7:8	$VD_{D/ASTM} = 0.36 + 1.12\ VD_{E/ASTM}$	0.98
2:11	$LD_{B/ASTM} = 6.67 + 0.68\ MD_{Sp8}$	0.95	7:9	$VD_{D/ASTM} = 2.67 + 0.63\ ID_{Bs}$	0.89
3:4	$LD_{C/ASTM} = -2.15 + 0.91\ LD_{D/ASTM}$	0.96	7:10	$VD_{D/ASTM} = 4.67 + 0.55\ MD_{Sp8}^{*}$	0.76
3:5	$LD_{C/ASTM} = 2.12 + 0.65\ LD_{Sp8}^{*}$	0.98	7:11	$VD_{D/ASTM} = 3.95 + 0.36\ MD_{Sp8}$	0.79
3:6	$LD_{C/ASTM} = 2.32 + 1.03\ LD_{Sp8}$	0.96	8:9	$VD_{E/ASTM} = 2.20 + 0.55\ ID_{Bs}$	0.89
3:7	$LD_{C/ASTM} = 3.88 + 1.70\ VD_{D/ASTM}$	0.98	8:10	$VD_{E/ASTM} = 3.96 + 0.48\ MD_{Sp8}^{*}$	0.75
3:8	$LD_{C/ASTM} = 4.21 + 1.95\ VD_{E/ASTM}$	0.98	8:11	$VD_{E/ASTM} = 3.25 + 0.32\ MD_{Sp8}$	0.80
3:9	$LD_{C/ASTM} = 7.87 + 1.13\ ID_{Bs}$	0.92	9:10	$ID_{Bs} = 2.60 + 0.95\ MD_{Sp8}^{*}$	0.92
3:10	$LD_{C/ASTM} = 11.10 + 1.03\ MD_{Sp8}^{*}$	0.82	9:11	$ID_{Bs} = 1.75 + 0.59\ MD_{Sp8}$	0.92
3:11	$LD_{C/ASTM} = 9.65 + 0.68\ MD_{Sp8}$	0.86	10:11	$MD_{Sp8}^{*} = 0.69 + 0.61\ MD_{Sp8}$	0.97
4:5	$LD_{D/ASTM} = 6.74 + 0.64\ LD_{Sp8}^{*}$	0.91			

Table 4. Variation and standard deviation for basalt and limestone.

Test	Variation	Standard Deviation	
		Basalt	Limestone
1	0.40	0.40	0.69
2	0.27	0.23	0.42
3	0.28	0.10	0.27
4	0.54[a]	0.16	0.46
5	0.33	0.45	0.23
6	0.42	0.41	0.19
7	0.35	0.17	0.19
8	0.21	0.09	0.24
9	0.68	1.52	1.36
10	0.75	0.86	0.33
11	0.97	0.64	0.94

[a]For 2 aggregate types, individual values were found with uncontrollable variations.

Figure 7. Mean of laboratory tests with limestone and basalt.
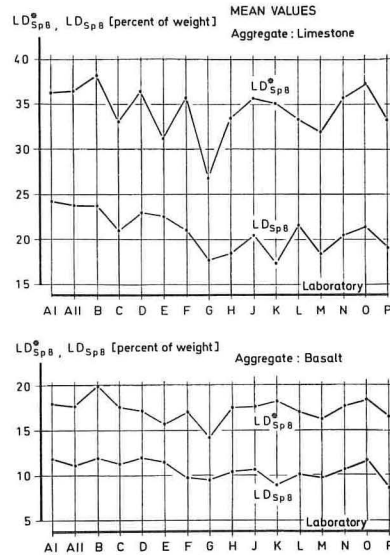
Table 5. Standard deviation and reproducibility under identical and comparative conditions.

Aggregate	Test	Standard Deviation		Reproducibility	
		Identical Conditions	Comparative Conditions	Identical Conditions	Comparative Conditions
Basalt	5	0.468	1.242	0.75	3.52
	6	0.415	1.037	0.66	2.95
Limestone	5	0.973	2.754	1.56	7.79
	6	0.454	2.180	0.73	6.08

Table 6. Requirements for small-sized coarse aggregates.

Courses	United States	France	Germany
Surface courses			19.2
Binder courses			
With high content of voids	40	20	19.2
With low content of voids			25.1
Bituminous mix base courses	45	25	29.5
Aggregate base courses	50	25(35)	25.1

The test values of the Deval test tend to be low, and the relative standard deviations are large. Therefore, no significant differentiation of abrasive and nonabrasive aggregates was achieved.

The Los Angeles test showed small standard deviations under identical conditions, and an international comparative test was performed to calculate standard deviations under comparative conditions (between laboratories).

The following 16 laboratories in 8 countries participated in this comparative test:

1. Tie-Ja Vesirakennushallitus, TVHin Laboratorio, Helsinki, Finland;
2. Valtion Teknillinen Tutkimuslaitos, Staatl. Techn. Forschungsanstalt, Helsinki, Finland;
3. Statens Vegvesen, Vegdirektoratet, Oslo, Norway;
4. Statens Väginstitut, Stockholm, Sweden;
5. Department of Civil Engineering, University of Salford, Salford, Great Britain;
6. MAPAG, Materialprüfungs-AG, Guntramsdorf, Austria;
7. Gumpoldskirchner Kalk- und Schotterwerke, Gumpoldskirchen, Austria;
8. Laboratoire Central des Ponts et Chaussées, Paris, France;
9. A branch laboratory of the preceding;
10. Evm. Szilikatipari Központi Kutato es Tervezö Intezet, Budapest, Hungary;
11. Bundesanstalt für Strassenwesen, Köln-Raderthal, West Germany;
12. Institut für bit. Baustoffe, TU München, München, West Germany;
13. Forschungsgemeinschaft Eisenhüttenschlacke, Rheinhausen, West Germany;
14. Institut für Baumaschinen, TH Aachen, Aachen, West Germany;
15. Ingenieurbüro Brand und Nies, Köln-Braunsfeld, West Germany; and
16. Institut für Strassenwesen, TH Aachen, Aachen, West Germany.

We used 2 different aggregates, basalt and limestone, and tested them with the 2 modified Los Angeles test methods, No. 5 and No. 6. The laboratories performed only the stress test with the prepared sample. The sieve analysis for calculating the test values was done in our laboratory. Thereby it was possible to find out the influence of the different Los Angeles machines on test results. Three samples were tested under the same conditions, so that every laboratory received 12 samples (3 basalt for No. 5, 3 basalt for No. 6, 3 limestone for No. 5, and 3 limestone for No. 6). The mean values of the 4 different tests obtained from the 16 laboratories are shown in Figure 7.

The values obtained at the different laboratories differ very much although their test procedures were conducted according to the standardized methods. With an analysis of variance (9) the variation within sets and between sets could be calculated (Table 5).

The standard deviations under identical conditions are within laboratories and those under comparative conditions are between laboratories. Table 5 also gives the reproducibility under identical conditions, $w = 1.96\sqrt{2}\,(s_o/\sqrt{n})$, and under comparative conditions, $v = 1.96\sqrt{2}\sqrt{s_1^2 + (s_o^2/n)}$, for a probability of 95 percent.

The reproducibility under comparative conditions was obtained in a range of about 20 to 30 percent, relative $(v/\bar{x})$, a range similar to the one we had found for the German impact test (19). This means that the Los Angeles test shows about the same variances under comparative conditions (comparing results of different laboratories) as the German impact test. To reduce this variability will require further standardization of the Los Angeles machine (length and diameter, slope, and steel) and the testing procedure. In that way we are now improving our German impact machine in order to reduce the variance of test values.

CONCLUSIONS AND APPLICATION

Linear regressions with high reliability among the Los Angeles, Deval, German impact, British impact, and modified Marshall impact tests were found. This means that, although the strains of the aggregates are very different with these test methods, they characterize the same or at least similar properties of aggregates. We learned that the magnitude of the test value is test specific but the quality of test results is aggregate specific.

In calculating the variances of the different test values, we found that the variation under identical conditions was small, especially for the Los Angeles test. However, comparing results under comparative conditions obtained in 16 different laboratories, we found that the Los Angeles test showed a reproducibility of about 20 to 30 percent relative, a range equal to or even larger than that found for the German impact test. This means that further standardization of testing machinery and of procedure is necessary if values of different laboratories are to be compared.

The calculated linear regressions found for the different test procedures will allow the comparison of specifications for small-sized coarse aggregates tested with different international test methods. It will now be possible to exploit international experiences and literature on the question of requirements for use of aggregates in road construction for different courses.

Table 6 gives a comparison of Los Angeles abrasion values that are required in the United States (34), France (35), and Germany (31). The German values that are required with the impact test, $\overline{SZ}_{Sp}$ (33), are transformed into Los Angeles values grading B (23) with the regression equations given in Table 3 (2:6) and shown in Figure 2.

The comparison shows that the requirements in France and Germany are similar, whereas the requirements of the United States are extremely broad. This may show how subjective requirements are fixed in different countries.

The calculated regressions will allow further exploitation of international experiences with aggregates of different qualities.

ACKNOWLEDGMENT

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GLASS BEADS IN PAINT

Carl F. Crumpton and George A. McCaskill, State Highway Commission of Kansas

Several items of laboratory equipment were used to study glass beads in paint. Wear and abrasion of beads and paint were studied with a type of surface abrasion equipment developed by California. Texture of road surfaces was measured by the use of cores and linear traverse equipment. Binocular and petrographic microscopes were used to study paint and bead problems in the field and laboratory. Normal headlight angles were simulated with a vertical illuminator on a microscope. Internal fractures in beads are more detrimental to light retroreflection than are bubbles, impurities, or nonround beads. Kansas glass bead specifications have been changed and have resulted in a lower unit price.

•THE OBJECTIVE of this overall study was to determine economical paint and glass-bead application rates for reflectorized traffic control stripes in Kansas. Results of the 1967 and 1968 field studies have been reported (12, 13). One significant observation of the field studies was that paint loss in Kansas was primarily due to chipping, not wear. A binocular microscope was so useful in the field studies that binocular stereo-zoom and trinocular petrographic microscopes were used in the laboratory bead and paint study. The petrographic microscope had a vertical illuminator, mechanical stage, and universal stage attachments.

This report deals with the various ways that the microscopes and other laboratory equipment were used to study glass beads in paint (5). Features of the paint and bead combinations were observed that helped explain field performance. Many samples were prepared specifically for laboratory studies, but several samples of paint stripes were removed from road surfaces for a microscopic study of paint and beads.

USE OF MICROSCOPE IN STUDYING FIELD PROBLEMS

Dulling of Stripes

In our early studies, transverse stripes were utilized for comparison of different paints and different bead gradations. Some stripes placed on concrete pavement showed a slight dulling over the hot-poured joint filler after a few weeks. This was at first interpreted to be due to bleeding of the joint filler material through the paint stripes. The nighttime appearance of these areas, however, was not diminished and, in fact, seemed brighter than the rest of the same stripe. A microscope examination revealed that the joint sealer had expanded causing the paint over it to crack (Fig. 1). The cracks were small and could not be readily seen without magnification. The effect of a series of subparallel microscopic cracks, however, was a slight but noticeable daytime dulling of the stripe over the joint filler. The increase in nighttime visibility over the joint filler was due to the exposure of the full sides of beads that were adjacent to cracks. Enhanced reflection is usually short-lived because as the filler expands it exudes from the joint and breaks through the paint stripe (13).

In another instance, centerline stripes on bituminous pavement patches were observed to be dulling prematurely. Microscopic examination revealed that asphalt was bleeding through the paint or being tracked onto it. Samples of the paint stripes were removed for study. They were sawed vertically through the layers of paint, and the sawed surfaces were polished on a lap wheel.

Microscopic study of the polished surface showed that the paint had many horizontal and vertical cracks that were filled with asphalt (Fig. 2). The asphalt was bleeding through cracks in the paint. The section of road in question, however, was scheduled for a new maintenance seal. After the seal was applied, no bleeding occurred, and the new stripes did not get prematurely dull.

Thick Buildup of Old Paint

During the microscope study of the asphalt-bleeding problem, we noted that several layers of paint were present. By observing a rough fractured surface through the paint, we found that the individual layers representing each striping operation during previous years could be studied. In this cross section of paint stripe, we could see the embedment of beads in each paint layer and determine which beads were effective reflectors and which ones were completely buried in the layer. The stripes were repainted before the buried beads could be exposed. Dust and grime were seen between some paint layers but did not seem to prevent bond.

This study confirmed Dale's contention (7, 8) that for a given wet paint film thickness there is only one size of bead that will give optimum optical performance. Seven separate layers of paint were present in one cross section of stripe. Each layer contained many beads that were buried and never exposed. This is not too surprising because Kansas was using an 18-mil wet paint film. Kansas glass-bead specifications allowed more than 60 percent of the beads to be 18 mils or smaller in diameter. When these beads were used in an 18-mil paint film, only the other 40 percent had any chance to retroreflect. Dale (8) calculated that in common usage the exposed beads capable of retroreflecting amount to only about 30 percent of the total beads used. Bead counts made from the cross sections of several old paint layers in Kansas showed this was closer to 25 percent, but we used 18 mils of paint whereas Dale calculated from 15 mils.

Several pieces of traffic line, which we collected from the same geographic area, had 1 thin layer of paint. We carefully removed the layers of paint down to the thin layer. Most of the beads were buried in this layer to about 60 percent of their diameter and undoubtedly were retroreflective (5). The thinner layer of paint allowed more beads to be retroreflective. This observation reinforced our belief that we could use thinner paint stripes with fewer beads but still have a better reflecting line than by using 18 mils with 6 lb of beads. Field tests proved we were right (12, 13).

Lack of Reflectivity of New Stripes

We were asked why some newly painted stripes were not reflecting at night so well as usual. We microscopically examined the stripes and found an overspray of paint on many beads (Fig. 3). Too much spray pressure or painting into a heavy wind might cause overspray. The operator was using higher spray pressure than recommended by the manufacturer. When he reduced the pressure the problem was solved.

Loss of Beads Due to Mold Growth

A large number of beads were lost from one section of 18-month old paint stripes. Microscopic study revealed a mold growing in the empty bead sockets (Fig. 4). Several loose beads in the paint were pried out, and mold was present under the bead. We believe the mold growth caused loosening of the beads previously lost. This is not considered a problem because we observed the phenomenon only on one section of reasonably old paint. Most of our centerline stripes are repainted yearly, and we doubt that much mold would grow in the paint during this time. Mold was observed in the lower part of the thick buildup of old paint layers previously discussed. Mold may contribute to the adhesion loss of paint stripes inasmuch as growth was observed in a number of old paint layers from several locations. The newer, upper paint layers seldom exhibited mold growth.

USE OF MICROSCOPES IN LABORATORY STUDIES

During the field studies of paint and beads (12, 13), we made a number of different types of laboratory studies. They were conducted to give information that could not be derived from field studies alone. Wet paint films from less than 5 to about 30 mils were placed on 4- by 8-in. glass plates. We dropped glass beads of many different gradations and application rates into the wet paint. Narrow strips $\frac{1}{4}$ to 1 in. wide were cut transversely from the larger plates for microscopic study. Glass and metal plates were used to obtain paint and bead samples as they were placed on the road. These too were used in the microscopic studies. The larger plates were also used for nighttime viewing and were illuminated by a single headlight beam at normal road angles.

Simulation of Headlight Angles

Figure 5 shows the microscope equipped with a vertical illuminator. The glass slide with paint and beads is standing on edge in this picture such that a thin beam of light from the illuminator can impinge on the glass beads at the same small angles that headlight beams are effective in nighttime driving. According to Dale (7), the angle is small, being only 1 deg, 14 min at 100 ft and 0 deg, 21 min at 350 ft in front of the vehicle.

The researcher can focus on one single bead to determine its light-reflecting properties. By reducing the magnification slightly, he can view a few beads at one time; by lowering the magnification considerably, he can view many beads at one time and compare the light-reflecting properties and depths of embedment of the various beads with each other.

There are many possible pitfalls in interpreting the results. For instance, the 3 beads shown in Figure 6 appear to reflect light differently depending on the amount of bubbles present. The bead on the left apparently contains the most bubbles and reflects the least light. The center bead contains a few bubbles and reflects light intermediate to the amount reflected by the other 2 beads. This would seem to demonstrate that air bubbles in the beads diminish their light-reflecting properties. Our studies, however, have shown that the amount of the bead exposed usually has more effect on its retroreflection than bubble inclusions (this assumes that the back of the bead is covered with paint to more than 50 percent of its diameter). In Figure 6, the bead that reflects the most light has its entire side exposed to the light beam and this is why it reflects more light than the other 2 beads. The center bead has part of its bottom half exposed and, therefore, reflects more light than the left bead that has only its upper half exposed.

Figure 7 shows the effect of exposing more bead surface. The bead is about 85 percent buried in paint. About one-third of the buried portion is exposed on the left. On the right, most of the paint film has been removed from the front of the bead increasing its retroreflection by exposing more surface area. Some chipping of paint to expose the sides of beads can thus improve retroreflection.

The gas bubble on the surface of the bead shown in Figure 7 appears to be a small dark spot but does not prevent reflection. Gas bubbles reduce retroreflection roughly by the amount of the surface area they comprise. This is an estimate based on a study of thousands of beads with various sizes and numbers of gas bubbles and not an accurately measured quantity of light reflection. In fact, the terms reflection and retroreflection as applied to glass beads may be misnomers. A bead more or less lights up like a diffusion light globe. There is no distinct beam of light reflecting back from a bead.

Our microscope studies show that internal fractures affect retroreflection considerably (Fig. 8). The beads shown in the paint panel on the left have been heated and then dropped into iced water to create internal fractures (the same procedure used in making "fried marble" jewelry). The beads on the right have not been internally fractured. The nonfractured beads reflect light, but the fractured beads do not.

Shadow Effect of Large Beads

The microscope was used to study the effect that shadows cast by large beads have on the reflection from smaller beads (7, 8). In our study, the paint and bead slide was

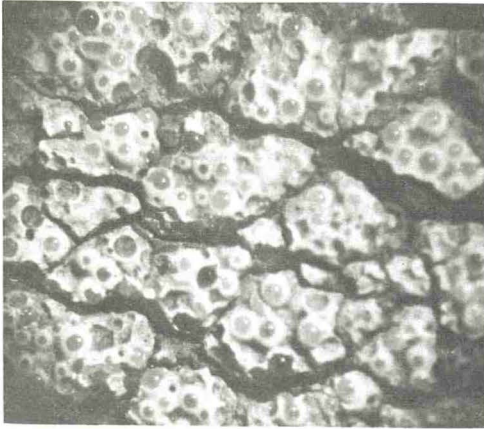Figure 1. Subparallel microscopic cracks in paint caused by expansion of joint filler.

Figure 2. Vertical cross section through several layers of glass-beaded paint stripe with asphalt-filled vertical cracks and subhorizontal planes.
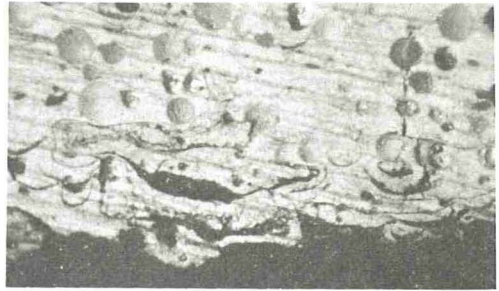

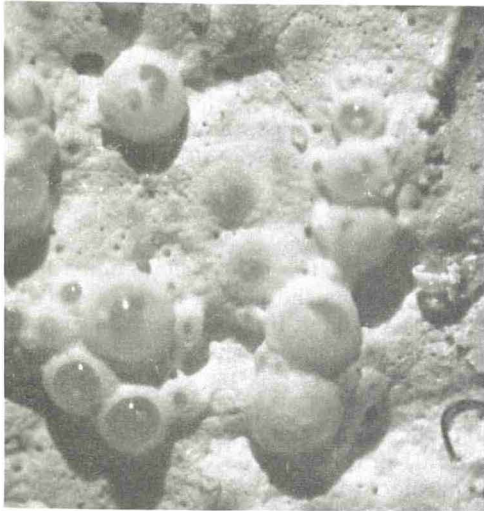Figure 3. Beads coated with paint because of too much spray pressure on paint gun.

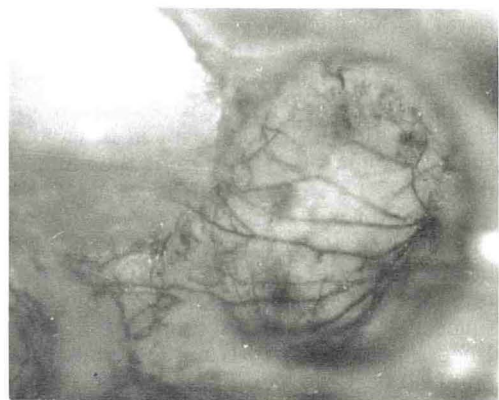Figure 4. Mold growth in empty bead socket.

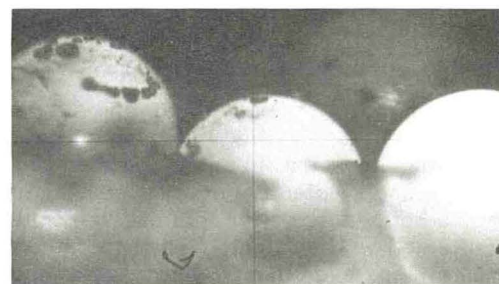Figure 5. Microscope with vertical illuminator.

Figure 6. Three beads that retroreflect different amounts of light.

laid flat on the stage of the microscope. The light was beamed in from the side at the approximate headlight angle. The researcher viewed the beads from above to see their back or shadow side. Many small beads fall in the shadows of larger beads and, thus, do not retroreflect (Fig. 9). The fact that beads cast shadows, however, indicates that they are retroreflecting. If light passed through a bead with no retroreflection, there would be little or no shadow. A bright spot at the back (left) of several beads shown in Figure 9 indicates that some light gets through.

The shadow effect is reduced when beads of only one size are used in a paint film that covers about 60 percent of the bead diameter. Using fewer beads reduces shadow problems too. Floating beads also reduce the shadow effect because of a more uniform size and because nearly half of each bead is exposed. The Colorado highway department (3, 4) reported benefits and savings when it used floating beads. Our studies indicate that the floating principle is important if a thick paint film is used. If, however, the wet thickness of the paint film is engineered to the diameter of a single-sized bead, then the results are much the same. The wet paint film thickness should cover 55 to 60 percent of the diameter of the beads used (6, 7, 8).

Beads in Black Paint

It has been stated that the bottom of a bead should not touch the pavement because the bead will reflect the pavement color, which, if it is black, will reduce the bead's visibility at night. We have not found this to be so. A bead is round and will rest on the pavement surface with only a small contact area at the very bottom of the bead. Pocock and Rhodes (15) show diagrams of the path of light entering a bead embedded in traffic paint. The rays of light focus on the back of the bead below the top paint line. The light rays do not refract to focus on the bottom of the bead. Dale (7, 8) photographed the back side of beads covered to various depths with paint. Dale's photographs showed the focal spot of light rays at the back of the beads, which indicated that the focusing diagrams of Pocock and Rhodes were likely correct.

In one of our studies we put beads into black paint. The beads appeared black when viewed directly from above (Fig. 10, left). When the panel was viewed at ordinary headlight angles, however, the beads reflected white (Fig. 10, right). Inasmuch as we did not foresee much benefit for Kansas, we did not pursue the research. If beads are found to reflect well in black paint, then states that use black lines between their white lines might wish to pursue the research and consider putting the beads in the black paint so that the white paint would remain cleaner and brighter in the daytime. Dale (7) stated that marking materials with dropped-on beads exhibit a far greater tendency to pick up road film than do unbeaded materials. These markings with heavy road film can be 100 percent intact and reflect at night yet fail in terms of delineation of the roadway in daytime. The road film would not show on black paint, and the unbeaded white lines would remain brighter for daytime use. We do not know why the beads reflected in black paint but wonder whether internal bubbles might act as small beads and actually enhance reflection in some cases. More research is needed on the reflectivity of beads in black paint to determine why they reflect for us but not for others.

OTHER STUDIES

Road Grime and Abrasion

Some paint stripes on new and old pavement sections in a city lost effectiveness rapidly during daylight but were satisfactory during nighttime. The city was subject to rather high traffic dust, and the roads were swept daily. We repainted the pavements and studied the new stripes. Less than 4 hours after placement, the stripes were noticeably duller. In one week the stripes had low daytime effectiveness but still reflected well at night. Using the microscope, we found traffic dust and grime covering the stripes even though they were swept a few minutes earlier. Figure 11 shows a 1-week old stripe. The brighter white spots are areas where the stripe was brushed with soap and water and then rinsed. A means of washing these stripes would improve their daytime effectiveness but perhaps only temporarily. The traffic grime and dust problem is usually worse in cities than in rural areas where faster traffic blows the dust away.

Paint and bead wear is increased if in areas similar to those just mentioned the traffic dust is coarse and harder than the beads. McCaskill and Crumpton (12, 13) reported paint and bead wear where sand- or gravel-surfaced roads intersected pavements with paint stripes. We studied this phenomenon in our laboratory by using a modified California surface abrasion device. Our asphalt research section uses it for evaluating slurry seal materials, but it has been used to study accelerated pavement wear (11) and paint stripes as well. Four-inch diameter concrete cylinders or Marshall asphalt plugs were painted and beaded on top for laboratory study. Field samples of 4-in. diameter pavement cores through a paint stripe can be used to study the pavement materials as well as the surface texture and beaded-paint layer. Figure 12 shows a beaded-paint film that has been subjected to 5 min in the abrasion machine. In addition to the rubber balls (17) used to evaluate asphalt slurry materials, a small amount of fine sand was added to simulate abrasive traffic material. Most of the beads abraded as badly as the paint. These surface-abraded beads are of little value as reflectors. The condition of the paint and beads shown in Figure 12 is almost identical to that of actual in-service paint stripes that are subject to abrasion.

Surface Profile Studies

Dale (7) discussed the importance of the pavement surface profile in bead retroreflection during rain. McCaskill and Crumpton (12, 13) reported that beaded-paint stripes on coarse seals were duller during dry weather but brighter at night during rain than comparable stripes on smooth seals. Our microscope studies showed that the beads did blank out when completely covered by water. To study the texture thoroughly, we made surface profiles across our paint stripes by using the device described by Dale (7), which we borrowed from him. It was satisfactory for field use and for many laboratory applications. In the overall surface texture studies, however, we were also interested in texture of trial asphalt slurry mixes in the laboratory. These materials were too soft for the mechanical surface profile recorder at the curing stage during which we wished to study them.

To overcome the problem, we utilized the motorized lathe and stereomicroscope portion of our linear traverse equipment. One focusing knob of the microscope was removed, and the shaft of a small rheostat used as a potentiometer was attached to the microscope focusing shaft. The rheostat body was affixed to the microscope so that the shaft was all that could move. During use a small constant voltage was fed into the rheostat, the output of which feeds through a preamplifier to a strip-chart recorder. The sample was placed on the lathe bed, and the equipment was referenced vertically and horizontally. The sample was then moved under the microscope just as in linear traverse work, and the operator kept the microscope in constant focus on the surface of the sample. As he focused up and down to compensate for differences in elevation of the surface, the voltage output from the rheostat was changed. This change was amplified and recorded. The end result was a tracing at several times vertical exaggeration of the surface texture.

This test is quite reproducible by different operators and was highly reproducible by one operator rerunning the same traverse. Figure 13 shows a trace of a paint surface on a 4-in. diameter core taken through a paint stripe on an in-service pavement. The trace is quite similar to those presented by Dale (7). The linear traverse method of surface profile, however, is not readily adaptable to field use. Cores of road surface must be obtained and brought to the laboratory for study. We have studied pavement surface texture in relation to skid properties with the equipment (11).

Raised Reflectors

Kansas has tried several types of raised reflectors for nighttime visibility during rain. The raised markers were plowed from the pavement during snowstorms. Chaiken (2) reported that some states prevented damage to raised markers by using rubber snowplow blades. Kansas tried rubber blades, but they were not suited to our average snowplowing conditions. The state has gone to carbide-tipped snowplow blades because they were the most suitable and economical of those tried.

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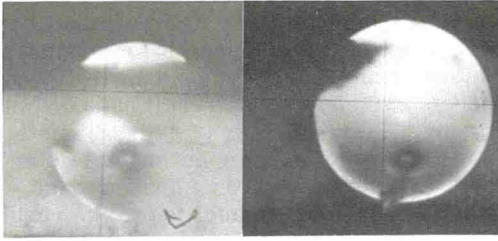Figure 7. Bead 0.012 in. wide buried in paint (left) and then exposed (right).

Figure 8. Beads with (left) and without (right) internal fractures.
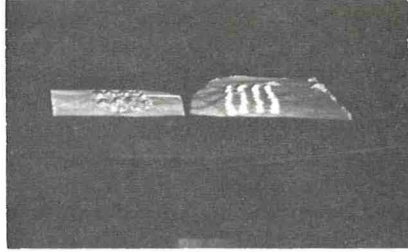

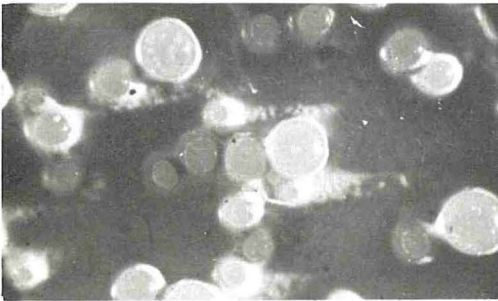Figure 9. Effect of shadows cast by large beads.

Figure 10. Beads in black paint where light strikes directly from above (left) and at average headlight angle (right).
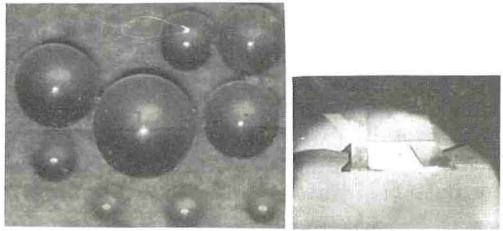

Figure 11. Week old paint stripe dulled in daylight by traffic dust and grime.
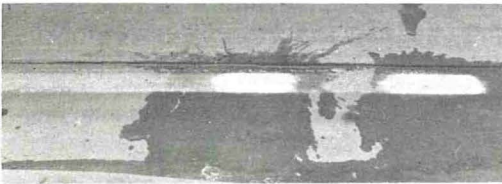

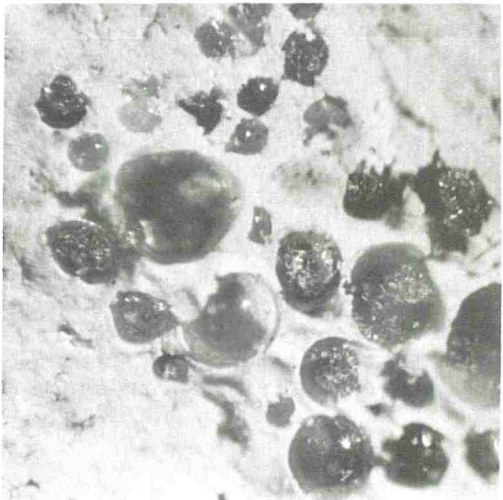Figure 12. Beaded-paint stripe abraded for 5 min.

Figure 13. Trace of painted surface of asphalt road ("valley" at arrow is slightly less than $\frac{1}{16}$ in. deep).

We have observed that grass growing in a crack between the pavement and a paved shoulder reflected light during a slow rain. Small rounded water droplets on the grass blades were responsible for the retroreflection. The water drops performed like glass beads. This observation leads us to wonder whether a system similar to synthetic turf material coated with paint and beads might work as raised reflectors that could "bend over" when necessary and withstand hard-surfaced, snowplow blade action. Posey (16) described a system of snow removal in which the blade rides above the pavement surface and the snow is lifted to the blade by air pressure. This system, if it is perfected, would be a boon to raised reflector use and snowplow blade life.

Intermittent Beading

Several years ago we placed three 18-in. long "pips" of beads on each 17.5-ft long paint stripe following the pattern suggested by Anderson (1). This worked satisfactorily, and the 3 beaded pips all blended together on the third stripe in front of a moving vehicle to appear as one fully beaded stripe. The intermittent pattern could be seen in the first and second stripe in front of the vehicle, but it was not distractive. A driver seldom looks at the first 2 stripes ahead; his attention has to be focused farther down the road. This research was not pursued, and our studies centered on using fewer beads and a thinner paint stripe. Partial beading has great potential, especially for edge lines, and merits more study.

Wicking of Paint on Beads

Many microscope pictures indicated that the paint wicked up the sides of the beads. Other samples from the same source of beads and paint, however, did not show the wicking effect. To study this discrepancy, we put beads in wet paint films of various thicknesses and watched the paint surface on the sides of the beads as the paint dried. No wicking was seen with our standard beads and paint.

The wicking appearance occurred only when the paint was thin and the beads large enough to drop to the bottom of the wet paint film and rest there with the bead protruding out of the paint film. The apparent wicking effect was due to the loss of volatiles and concurrent shrinkage of the paint film. The original top level of the paint film remained around each bead but as the paint dried the area between beads shrank and gave as the end result the false impression that the paint had wicked up the side of the beads. When thick films of paint were used and the bottom of the beads did not drop to the glass plate, there was no sign of wicking. In this case, as the paint shrank because of evaporation of solvent, the entire original surface, beads and all, settled to a lower elevation. Some paint and bead combinations did wick, and with a microscope the paint film could be seen moving up the side of the beads. This was very evident on some of the black paint we tried.

General Observations

During the microscope studies of glass beads in paint, a number of general observations were made that have not been covered elsewhere in this report. They will be listed here without much discussion although some have been discussed by other researchers.

Transmitted light was better for studying the internal conditions of glass beads (fractures, bubbles, and impurities) than reflected light. Switching from transmitted to reflected light for individual beads helps in studying how the internal conditions affect retroreflection. Beads with only a few bubbles and impurities were about as reflective as beads with no flaws, and in some cases small internal bubbles may have enhanced reflection. Slightly out-of-round beads reflected about as well as round beads. Very oblong beads gave some reflection but not so much as round beads unless the beam of light was parallel to the long axis. There are fragments and stringers of glass in many bead samples, and such particles do not reflect. Two or more beads fused together or beads with large protruding surface knobs generally reflect little light, but sometimes they reflect well. Most beads "blank out" when covered with a thin film of water, but a very few from each sample were reflective under water.

Studded tires, if they pass over a paint stripe, fracture some of the beads and render them useless. Studded-tire wear is being seen in Kansas, but the problem is less than in some states; however, the situation may get worse. Some Kansas drivers use their studded tires all year even though such use during the summer is unlawful.

An ordinary mirror can be used above the ocular of the microscope to project the image of the beads onto a movie screen for group viewing in a darkened room. A section of sieve screen cut from any desired size of screen stock along with Scotch tape as backing makes a handy bead holding and sizing device for microscope study. The tape adheres to the screen and sticks to the beads that are small enough to drop into the square holes of the sieve. Different sieve stock can be used for different-sized beads.

REMOVAL OF OLD PAINT BEFORE REPAINTING

One way that glass beads become ineffective is loss through chipping of a beaded-paint stripe. Chipping is the primary loss mechanism in Kansas (13). A suggestion for reducing the paint-chipping problem was to find a cheap, fast method of removing old loose paint before the pavement is repainted (13). We investigated various methods of paint removal under the following restrictions:

1. They had to be rapid and fit into the regular painting operation (more than 100 miles of painting in 1 day in some instances); and
2. The stripes removed had to be repainted the same day (safety standards in Kansas do not provide for allowing sections of centerline to go unpainted very long).

Heater units were too slow and unsuited to Kansas painting operations. Sandblasting was too slow and a hazard to traffic. Chemical removers were too slow, hazardous to workers, and harmful to the finish of cars. High-pressure water jets were too slow, hazardous to traffic, and required too much drying time before repainting could be done. Mechanical burring and brushing methods were too slow. All of these methods were too expensive.

The only technique we tried that was even partially successful under the restrictions listed was a jet of air directed at a low angle onto the paint stripe right ahead of the striper. This removed the loosest and thus most detrimental flakes of old paint. The air can be supplied by the striper if its compressor is sufficient to place the paint and beads and still have capacity to send a stream of air onto the old stripe. Most Kansas stripers cannot supply the extra air.

Inasmuch as Kansas now uses a thinner stripe and repaints less frequently, the paint buildup is less of a problem. If the chipping problem persists, however, a separate truck-mounted, high-capacity, high-pressure air supply unit to work ahead of the paint striper would be worthy of consideration. Posey (16) described a similar system for snow removal, and it might be engineered to remove loose paint.

Allowing more time to elapse before repainting and letting the loose paint be removed by traffic, wind, or rain were considered. Painting individual centerline stripes in the skip area between the old stripes was also considered. Another suggestion was to paint the minimum stripe width allowed and to repaint the new line alongside the old with about an inch of sidelap between them. These considerations were generally rejected, however, because of the probability of a less attractive finished stripe.

IMPLEMENTATION OF RESULTS

We have reported the previous implementation of results (13). We also recommended that the paint thickness be reduced to 10 mils with 4 lb of beads per gallon. This was based on field results and on laboratory studies described here. Kansas has implemented that recommendation.

As a result of our laboratory microscope studies of various bead gradations in paint films of different thicknesses, we agreed to a change in Kansas bead gradations to bring the bead sizes into a more compatible relation with our lesser paint film thickness. The numbers of large and small beads were both reduced.

Before the gradation changes were written into specifications, microscope slides and metal plates with 4-in. wide panels of the new gradation of beads in a 10-mil paint film

were prepared. We examined the slides by using the vertical illuminator and simulated headlight angles as shown in Figure 5. The larger plates of paint and beads were examined at distances as great as 200 ft in our laboratory after dark; a single headlight beam was used. Both the microscope and headlight studies indicated that the new bead gradation would provide good retroreflection in 10 mils of paint.

Inasmuch as we were making a gradation change that might be difficult or expensive to produce, our Materials Department contacted all the major glass-bead manufacturers to discuss the proposed changes. All agreed that the new gradation would present no problems to them. Since a change in specifications was being made, it was decided to look at other parts of Kansas glass-bead specifications also to see whether other changes might be worthwhile. The company representatives were each asked for their recommendations on other requirements that should be considered for change. In addition to the gradation changes, 14 other items were considered and more changes made. The number of imperfect beads allowed was changed; packaging and marking requirements were changed. Lot sizes for testing purposes were changed and resulted in less sampling and testing. The new specifications, No. TS 46.19, were issued on May 9, 1969, in time to make bead purchases for fiscal 1970.

We were warned that the proposed changes might raise the price, but instead a big reduction occurred. Even the price of the highest bidder was below the price we had been paying for beads. With the additional miles of edge lines being painted, a reduction in the cost of beads was indeed welcome.

In the earlier report, we stated that not all of the savings would show up in the records as less money spent on paint and beads (13). The savings in materials costs were used in large part to pay for many miles of additional edge-line striping. According to the data supplied to Permoda, Moore, and Chaiken (14), only 21 percent of the Kansas white paint striping in 1965 was edge lines. Now edge lines account for about 60 percent of the white paint striping. This additional edge-line striping is being paid for by the overall savings in material costs.

There has also been less spent for materials even though the unit cost of paint is up. Calculations from data presented by Permoda, Moore, and Chaiken (14) show that Kansas spent $668,582 on paint and beads placed on roads in 1965. Maintenance department records show that the materials used for striping in fiscal year 1969 cost $665,122 or $13,460 less than in 1965. Records for fiscal 1970 show $506,862 spent for the paint and beads used that year, $161,720 less than in 1965. The purchase requests for paint and beads for fiscal year 1971 amounted to $519,553, which is $149,029 less than 1965 usage. This brings the total 3-year savings to $324,209. In a period of spiralling inflation, such savings are rare and can be used to offset increasing prices in other operations.

Savings in materials costs are not the only benefit gained from the study. A new pride in the striping work has been established as a result of the attention given to it. The interest of the paint-striping crews has improved, and a competitive spirit has developed among them. New striping records have been set and broken several times (9) since the record was established of 92 miles of painting in one day (13). The latest Kansas record is 118.1 miles—41.3 miles of center stripes and 76.8 miles of edge lines (10). Other side benefits from the lesser paint and bead requirements is that overall there is less testing, less storage space, and less handling problems than would be involved with greater paint thickness and bead application rates. Lesser amounts of materials used also mean less pollution. There is less solvent to escape into the atmosphere and less solid materials to flake and wear away to add to the world's waste products.

SUMMARY AND CONCLUSIONS

This report explains the practical use of several items of laboratory equipment to study the performance of glass beads in paint. Many aspects of the paint and bead problem were studied with the use of binocular and petrographic microscopes. Wear and abrasion of paint and beads were studied with the use of the California-developed surface abrasion equipment. Cores and linear traverse equipment were used to measure

the texture of road surfaces. The following observations and results are based on field and laboratory studies:

1. Microscopes are very useful in studying paint and bead problems both in the field and in the laboratory. A vertical illuminator can be used on a microscope to simulate normal headlight angles to study the performance of various bead sizes and gradations in paint films of differing thicknesses. Much of the engineering of glass-bead size and gradation to fit the wet film thickness of paint being used or anticipated for use can be done in the laboratory with the use of the microscope techniques described. Our glass-bead specifications were changed with such studies as a guide. This has resulted in a lower unit price for beads. Microscope studies indicate that internal fractures are more detrimental to light retroreflection than are bubbles, impurities, or nonround beads.

2. Glass beads in some black paints do reflect light.

3. New stripes that are dull in daytime may only be covered by traffic grime and dust. The dust problem is greater in cities than in rural areas because of the slower city traffic.

4. Kansas now uses 10 mils (wet) paint with 4 lb of drop-on beads per gallon of paint.

5. Painting is now faster than before, and one crew has placed more than 118 miles of beaded paint stripes in an 8-hour day.

6. This project, which cost less than $20,000 in research funds, has saved about $324,209 in paint and bead costs for fiscal years 1969, 1970, and 1971 compared with 1965. In addition, the cost of the materials for many additional miles of edge-line painting has been paid for by the implemented changes.

7. Savings in storage space, testing, and handling of paint and beads have also occurred.

8. A stream of air blown over an old stripe just ahead of the paint striper removes the loosest and most detrimental flakes of old paint and, therefore, is somewhat effective in increasing paint and bead life. Other attempts to rapidly remove the old paint prior to repainting have been largely negative.

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